



EDGEWOOD

CHEMICAL BIOLOGICAL CENTER

J.S. ARMY RESEARCH, DEVELOPMENT AND ENGINEERING COMMAND

ECBC-TR-778

MATERIAL DEMAND STUDIES: MATERIALS SORPTION OF VAPORIZED HYDROGEN PEROXIDE

Lawrence R. Procell
Zoe A. Hess
David G. Gehring
Joseph T. Lynn
Philip W. Bartram
Teri Lalain

RESEARCH AND TECHNOLOGY DIRECTORATE

Shawn Ryan
Brian Attwood
Blair Martin



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Washington, DC 20460

Mark D. Brickhouse

Booz | Allen | Hamilton

BOOZ | ALLEN | HAMILTON
Belcamp, MD 21017-1535

June 2010

Approved for public release;
distribution is unlimited.



ABERDEEN PROVING GROUND, MD 21010-5424

Disclaimer

The findings in this report are not to be construed as an official Department of the Army position unless so designated by other authorizing documents.

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing this collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. **PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.**

1. REPORT DATE (DD-MM-YYYY) XX-06-2010		2. REPORT TYPE Final		3. DATES COVERED (From - To) Nov 2003 – Jul 2005													
4. TITLE AND SUBTITLE Material Demand Studies: Materials Sorption of Vaporized Hydrogen Peroxide				5a. CONTRACT NUMBER													
				5b. GRANT NUMBER													
				5c. PROGRAM ELEMENT NUMBER													
6. AUTHOR(S) Procell, Lawrence R.; Hess, Zoe A.; Gehring, David G.; Lynn, Joseph T.; Bartram, Philip W.; Lalain, Teri (ECBC); Ryan, Shawn; Attwood, Brian; Martin, Blair (EPA); and Brickhouse, Mark D. (Booz Allen Hamilton).				5d. PROJECT NUMBER EPA IAG DW 939917-01-0													
				5e. TASK NUMBER													
				5f. WORK UNIT NUMBER													
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) DIR, ECBC, ATTN: RDCB-DRP-D//RDCB-DRB-D, APG, MD 21010-5424 EPA, 1200 Pennsylvania Ave, N.W., Washington, DC 20460 Booz Allen Hamilton, 4692 Millennium Dr, Ste. 200, Belcamp, MD 21017				8. PERFORMING ORGANIZATION REPORT NUMBER ECBC-TR-778													
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) EPA, 1200 Pennsylvania Avenue, N.W., Washington, DC 20460				10. SPONSOR/MONITOR'S ACRONYM(S) EPA													
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)													
12. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.																	
13. SUPPLEMENTARY NOTES																	
14. ABSTRACT: <p>To address Homeland Security's needs for decontamination, the EPA established an Interagency Agreement with the U.S. Army Edgewood Chemical Biological Center (ECBC) to take advantage of ECBC's extensive expertise and specialized research facilities for the decontamination of surfaces contaminated with chemical and biological (CB) warfare agents. The National Homeland Security Research Center (NHSRC) collaborated with ECBC, in a mutual leveraging of resources, to expand upon ECBC's on-going programs in CB decontamination. The goal was to more completely address the parameters of particular concern for decontamination of indoor surfaces in buildings following a terrorist attack using CB agents, toxic industrial chemicals (TICs), or materials (TIMs). Vaporized hydrogen peroxide (VHP[®]) and chlorine dioxide (ClO₂) are technologies that have been used to decontaminate indoor surfaces contaminated with anthrax. The technologies also show potential for use in decontaminating indoor surfaces contaminated by chemical agents. This program is specifically focused on decontamination of the building environment to restore a public building to a usable state after a terrorist contamination episode. Because building interiors may contain a large surface composed of concrete cinder block, wood, steel, carpet, ceiling suspension tile, and painted wallboard, the effort was designed to determine how building materials impact the concentration/titer of decontaminant in the vapor phase. The focus of this technical report is the evaluation of the building interior materials and the Steris VHP technology.</p>																	
15. SUBJECT TERMS <table border="0"> <tr> <td>Vaporized hydrogen peroxide</td> <td>VHP</td> <td>EPA</td> <td>Building materials</td> </tr> <tr> <td>Steris Technology</td> <td>Material demand</td> <td>Fumigant</td> <td>Biological agents</td> </tr> <tr> <td>Drager Electrochemical Detector</td> <td>Building restoration</td> <td>NHSRC</td> <td></td> </tr> </table>						Vaporized hydrogen peroxide	VHP	EPA	Building materials	Steris Technology	Material demand	Fumigant	Biological agents	Drager Electrochemical Detector	Building restoration	NHSRC	
Vaporized hydrogen peroxide	VHP	EPA	Building materials														
Steris Technology	Material demand	Fumigant	Biological agents														
Drager Electrochemical Detector	Building restoration	NHSRC															
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON Sandra J. Johnson												
a. REPORT U	b. ABSTRACT U	c. THIS PAGE U			19b. TELEPHONE NUMBER (include area code) (410) 436-2914												

20100624421

Blank

PREFACE

To address Homeland Security's needs for decontamination, the U.S. Environmental Protection Agency (EPA) established an Interagency Agreement with the U.S. Army Edgewood Chemical and Biological Center (ECBC) to take advantage of ECBC's extensive expertise and specialized research facilities for the decontamination of surfaces contaminated with chemical biological (CB) warfare agents. The EPA's National Homeland Security Research Center (NHSRC) collaborated with ECBC to determine the impact of vaporized hydrogen peroxide (VHP) as a decontaminant on indoor surfaces in buildings. The VHP work was completed under EPA IAG DW 939917-01-0. The work was conducted from November 2003 to July 2005 and recorded in ECBC laboratory notebook numbers 05-0059, 04-0043, and 05-0044.

This report was published through the Technical Releases Office; however, it was edited and prepared by the Decontamination Sciences Branch, Research and Technology Directorate, ECBC.

The use of either trade or manufacturers' names in this report does not constitute an official endorsement of any commercial products. Manufacturer names and model numbers are provided for completeness. This technical report may not be cited for purposes of advertisement.

This report has been approved for public release. Registered users should request additional copies from the Defense Technical Information Center; unregistered users should direct such requests to the National Technical Information Service.

Acknowledgments

A program cannot be successfully completed without the contributions of a good team of people. The authors thank the following individuals for their contributions toward the successful completion of this test program:

- Dave Stark for completing the independent audits per the EPA program requirements.
- Dr. David Cullinan for preparing many coupon run baskets, performing coupon measurements, and preparing chain-of-custody forms during the time his assigned laboratory was closed.
- Dave Sorriek for assistance with acquiring materials and equipment fabrication.
- Diane Simmons for assistance with the preparation of this report.
- Drs. Emily Snyder and Zhishi Guo, and Leroy Mickelsen for reviewing and commenting on the final report.
- Diane Wylie for the technical review and preparation of this report.

Blank

CONTENTS

1.	BACKGROUND	1
2.	SUMMARY OF CONCLUSIONS.....	1
3.	INTRODUCTION	2
3.1	Dehumidification	3
3.2	Conditioning.....	4
3.3	Decontamination	4
3.4	Aeration.....	4
4.	EXPERIMENTAL PROCEDURE	4
4.1	Representative Building Material Test Coupons	4
4.2	Hydrogen Peroxide Test Chamber	6
4.3	Material Demand Testing.....	7
4.3.1	Dehumidification	8
4.3.2	Conditioning	8
4.3.3	Decontamination.....	8
4.3.4	Aeration	9
4.4	Data Review and Technical Systems Audits.....	9
4.5	Material Demand Calculation	9
5.	EVALUATION OF EMPTY CHAMBER	10
5.1	“Fog” Test Results and Discussion	10
5.2	Baseline Tests and Results	11
5.3	Baseline Test Discussion.....	15
6.	EVALUATION OF BUILDING MATERIALS	16
6.1	Results	16
6.2	Discussion	31
6.3	Consequences for Building Decontamination	32
7.	QUALITY ASSURANCE FINDINGS	34
8.	CHALLENGES AND LESSONS LEARNED.....	34
8.1	VHP Relative Humidity Sensors.....	34
8.2	Calibration of Dräger Hydrogen Peroxide Electrochemical Sensors	35
	LITERATURE CITED.....	37
	ACRONYMS.....	39
	APPENDIXES:	
A.	DETAILED COUPON PREPARATION AND INSPECTION PROCEDURES	41
B.	COUPON IDENTIFIER CODE	45

FIGURES

1.	The Steris VHP decontamination cycle.....	3
2.	Samples of the test coupons.....	5
3.	ECBC exposure chamber.....	6
4.	Exposure chamber fog test.....	11
5.	Illustration of the determination of zero time.	12
6.	Baseline VHP exposure tests.	13
7.	Concentration profile for baseline VHP tests.	14
8.	VHP process using a baseline test.	15
9.	Chamber temperature profile throughout testing.....	16
10.	Representative VHP test results for carpet.	18
11.	Representative VHP test results for steel.....	19
12.	Representative VHP test results for painted gypsum wallboard.....	20
13.	Representative VHP test results for acoustical ceiling tile.	21
14.	Representative VHP test results for wood.	22
15.	Representative VHP test results for concrete cinder block.....	23
16.	Representative concentration profile results for carpet.	25
17.	Representative concentration profile results for steel.....	26
18.	Representative concentration profile results for gypsum wallboard.....	27
19.	Representative concentration profile results for acoustical ceiling tile.	28
20.	Representative concentration profile results for wood.	29
21.	Representative concentration profile results for concrete cinder block.....	30
22.	Aeration time for building materials exposed to 250-ppmv VHP.	32
23.	Comparison of VHP concentration and %RH.	35
24.	IOP DS04016 Coupon placement in chambers.	46

TABLES

1.	Representative building interior materials.....	4
2.	Coupon exposed area.....	8
3.	Baseline material demand test results.....	12
4.	Material demand results for building materials (125–150 ppmv).	24
5.	Material demand results for building materials (250–300 ppmv).	24
6.	Material demand of warehouse surfaces.....	33
7.	Material demand of office surfaces.	33

MATERIAL DEMAND STUDIES: MATERIALS SORPTION OF VAPORIZED HYDROGEN PEROXIDE

1. BACKGROUND

The Material Demand effort was designed to determine how building materials impact the ability to maintain a target decontaminant vapor concentration within an enclosed interior space. The building materials may impact the decontaminant vapor concentration by either sorption or decomposition of the decontaminant. Since building interiors may contain large surface areas consisting of different materials, data are needed to determine how these interior surfaces affect the ability to maintain a stable target concentration. Vaporized hydrogen peroxide (VHP[®]) and chlorine dioxide (ClO₂) were selected because these decontamination technologies have been used to decontaminate indoor surfaces contaminated by anthrax and/or show potential for use in decontaminating indoor surfaces contaminated by chemical agents. The VHP results are presented in this report. The representative building interior materials tested were unpainted concrete cinder block, standard stud lumber (wood 2 x 4 in., fir, type-II), latex-painted 0.5 in. gypsum wallboard, ceiling suspension tile, painted structural steel, and carpet.

2. SUMMARY OF CONCLUSIONS

- The VHP Material Demand tests showed that the building materials affect the VHP decontamination vapor concentration. The impact varies based on the type of material.
 - The concrete cinder block coupon had the greatest impact on maintaining the VHP concentration due to decomposition of the VHP.
 - The cellulose-based materials, wood and ceiling tile, showed adsorption of the VHP with a high material demand value, which was approximately one-third to one-half the value for concrete. VHP desorption resulted in a long aeration time.
 - The wallboard had a moderate effect on the VHP concentration compared with the concrete cinder block.
 - The carpet and steel coupons had a low impact on the VHP concentration compared with the concrete cinder block.
- The relative humidity sensor measurements were affected by the presence of VHP. The sensor read high in the presence of VHP, but responded normally with no apparent visual degradation after the VHP was removed.
- The hydrogen peroxide sensor performance verification, using the wet-chemical titration method, showed that both the inlet and outlet sensors were not adversely affected by prolonged and repeated exposure to VHP. Neither of the two sensors showed evidence of visual deterioration or change in response during the testing period.

- Hydrogen peroxide concentration sensor calibration, using sulfur dioxide in nitrogen gas, revealed that the sensors were highly sensitive to changes in pressure. For the custom-built exposure chambers, inline sensor calibration was recommended.
- The percentage of VHP decomposition in the chamber was a function of VHP/airflow through the chamber. A VHP/airflow of 3.0 ft³/min was used in all tests. Increasing the flow above 3.0 ft³ / min significantly changed the peroxide loss.

3. INTRODUCTION

To address Homeland Security's needs for decontamination, the U.S. Environmental Protection Agency (EPA) established an Interagency Agreement with the U.S. Army Edgewood Chemical and Biological Center (ECBC) to take advantage of ECBC's extensive expertise and specialized research facilities for the decontamination of surfaces contaminated with chemical and biological (CB) warfare agents. The EPA National Homeland Security Research Center (NHSRC) collaborated with ECBC in a mutual leveraging of resources, to expand upon ECBC's on-going programs in CB decontamination. The goal was to more completely address the parameters of particular concern for decontamination of indoor surfaces in buildings following a terrorist attack using CB agents, toxic industrial chemicals (TICs), or materials (TIMs). Vaporized hydrogen peroxide (VHP[®]) and chlorine dioxide (ClO₂) are decontamination technologies that have been used to decontaminate indoor surfaces contaminated with anthrax. The technologies also show potential for use in decontaminating indoor surfaces contaminated by chemical agents. This program is specifically focused on decontamination of the building environment, to restore a public building to a usable state after a terrorist attack. Systematic testing of decontamination technologies generates objective performance data so building and facility managers, first responders, groups responsible for building decontamination, and other technology buyers and users can make informed purchasing and application decisions.

Since building interiors contain a large surface composed of different materials, the Material Demand effort was designed to determine how building materials impact the concentration of decontaminant in the vapor phase. The objective of this study was to establish and conduct laboratory test procedures to determine to what degree building materials affect the vaporized decontaminants. The building interior materials used for testing are a subset of the variety of structural, decorative and functional materials common to commercial office buildings regardless of architectural style and age. The building materials encompass a variety of material compositions and porosities. The materials studied included unpainted concrete cinder block, standard stud lumber (wood, 2 x 4 in. fir), latex-painted 0.5 in. gypsum wallboard, acoustical ceiling suspension tile, primer-painted structural steel, and carpet. The focus of this technical report is the evaluation of the building interior materials and VHP.

The VHP[®] technology developed by Steris (EPA registration #58779-4) has been in use for more than a decade. The VHP fumigant was initially used to sterilize pharmaceutical processing equipment and clean rooms.^{1,2} In response to the anthrax attacks of October 2001, Steris adapted its VHP technology to perform the decontamination of two U.S. government facilities—the General Services Administration (GSA) Building 410 at Anacostia Naval Base in Washington DC and the U.S. Department of State SA-32, Sterling, VA mail center. Through a joint venture with Steris Corporation, the application of VHP and modified VHP for chemical- and biological-agent decontamination have been successfully tested in laboratory, large-chamber, and field demonstrations,

including a former office building and a C141 aircraft.³⁻⁵ VHP also reacts with and neutralizes VX and HD chemical agents.⁶

Decontamination of an interior space using VHP is a four-phase process involving preparation of the building interior air (dehumidification), achieving a steady state decontaminant level (conditioning), performing the decontamination, and then aerating for safe reentry (Figure 1).³

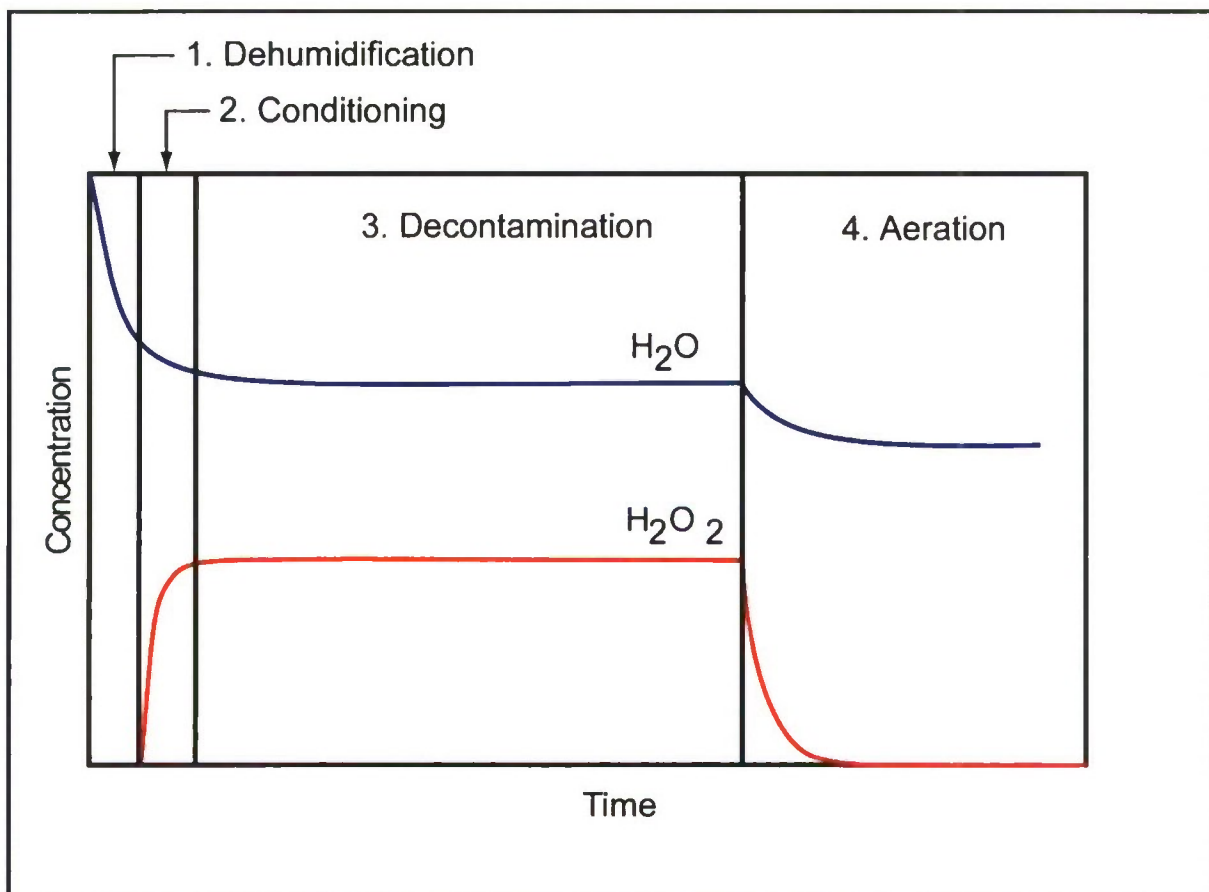


Figure 1. The Steris VHP decontamination cycle.

3.1 Dehumidification

Hydrogen peroxide vapor can co-condense with water vapor, producing an undesired condensate high in hydrogen peroxide. If ambient conditions are likely to permit condensation—high humidity and/or cold temperatures—this can be prevented by circulating dry, heated air through the interior prior to injection of the hydrogen peroxide vapor. The target humidity level was determined by the concentration of vapor to be injected and the desired steady state concentration for the decontamination. The lower relative humidity permits a higher concentration of hydrogen peroxide without reaching a saturation point. For this study, the maximum relative humidity at start-of-run (prior to introducing decontaminant) was 30%.

3.2 Conditioning

During the conditioning phase, the injection of hydrogen peroxide vapor was initiated at a rapid rate to reach the desired concentration set point without condensation. Once the target concentration was achieved, the injection rate was lowered to maintain the set-point concentrations.

3.3 Decontamination

Decontamination is a timed phase, which is dependent on the hydrogen peroxide vapor concentration. In building and aircraft applications, a decontamination timer counts down from the preset decontamination time. If the concentration or temperature values fall below the set point, the timer stops. During the decontamination phase, this ensures that the interior space is exposed to at least the minimum decontamination conditions for the desired exposure time. For this laboratory-scale study, the enclosure VHP concentration was maintained uninterrupted, within the target concentration range.

3.4 Aeration

After completion of the decontamination phase, the hydrogen peroxide injection was terminated. Air was introduced into the chamber. The air displaced the hydrogen peroxide. The system was monitored until the hydrogen peroxide concentration fell to a safe level for coupon removal.

4. EXPERIMENTAL PROCEDURE

The Material Demand testing was conducted in compliance with the Quality Assurance Project and Work Plan (QAPP)⁷, developed under the Quality Management Plans (QMP)^{8,9} and EPA E4 quality system requirements.^{5,10-12}

4.1 Representative Building Material Test Coupons

Test coupons were prepared in accordance with the ASTM testing requirements for the Material Compatibility testing.¹³ The coupons were cut from stock material in accordance with the procedure in Appendix B of the QAPP⁷ and reproduced as described in Appendix A of this report. The coupons were cut from a large sheet of material, sufficient for obtaining multiple test samples with uniform characteristics (i.e., test coupons were all cut from the interior rather than the edge of a large piece of material). The building materials studied, including supplier and coupon dimensions, are provided in Table 1 and shown in Figure 2.

Table 1. Representative building interior materials.

Material	Code	Supplier	Length (in.)	Width (in.)	Thickness (in.)
Structural Wood, Fir	W	Home Depot	10.0	1.5	0.5
Latex-Painted Gypsum Wallboard	G	Home Depot	6.0	6.0	0.5
Concrete Cinder Block	C	York Supply	4.0	8.0	1.5
Carpet	R	Home Depot	6.0	8.0	0.0
Painted Structural Steel	S	Specialized Metals	12.0	2.0	0.3
			5.3	0.8	0.3
Ceiling Suspension Tile, Acoustical	T	Home Depot	12.0	3.0	0.6

Chain-of-Custody (CoC) cards were used to ensure traceability of the test coupons throughout all phases of testing. Before testing began, the coupons were measured to ensure that the acceptable tolerances were met (Appendix A). Coupons were also visually inspected for defects and/or damage. This information was recorded on the CoC card. Coupons that were not within the allowable size tolerances and/or damaged were discarded. Each coupon was assigned a unique identifier code matching the coupon with the sample, test parameters, and sampling scheme (Appendix B). The unique identifier code was recorded also on the CoC form. The CoC cards followed each sample from Material Demand testing, through Material Compatibility testing, to disposal.



*Coupons are not shown to scale

Figure 2. Samples of the test coupons.

4.2 Hydrogen Peroxide Test Chamber

A Plas-Labs compact glove box (Model 830-ABC), fitted with Hypalon[®] gloves and glove port plugs, was used as the exposure chamber (Figure 3). The glove box was acrylic with an internal volume of 11.2 ft³ (28 in. wide x 23 in. deep x 29 in. high) and an isolated transfer chamber that was 12 in. long x 11 in. diameter (I.D.). The chamber was insulated with 0.5 in. thick polyisocyanurate foam (R value 3.3) to help stabilize exposure temperature and minimize the possibility of VHP or water condensation (insulation not shown in Figure 3). The chamber insulation also blocked the exposure of VHP to light and minimized possible VHP decomposition. An exposure rack, constructed of Lexan[®] and horizontal stainless steel bars, was used to hold the test specimens. The exposure rack was 12 in. long x 12 in. wide x 24 in. high, and had four levels for specimens. Coupons were placed in the glove box in accordance with IOP DS04016 as shown in Figure 3 and Appendix B, Figure 24.

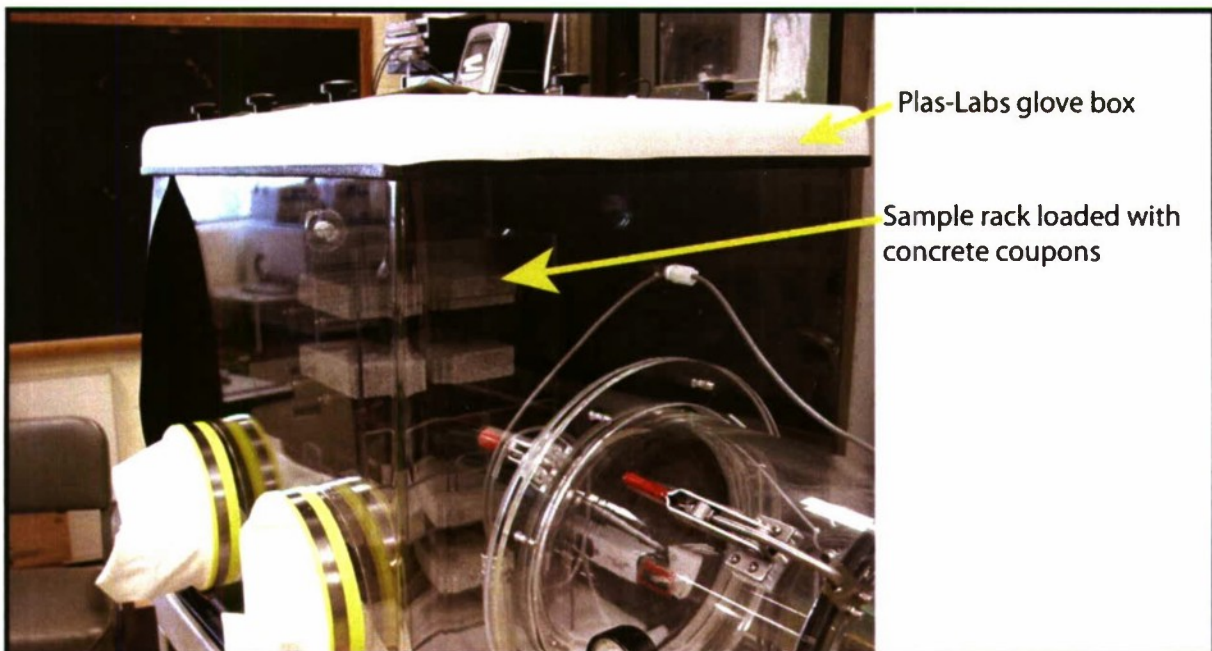


Figure 3. ECBC exposure chamber.

The vapor concentration, temperature, and relative humidity were recorded each minute during testing. The VHP concentrations were measured using two Dräger hydrogen peroxide electrochemical sensors (model HC 6809070), coupled with Dräger Polytron 2 transmitters for real-time monitoring at the inlet and outlet of the chamber. The sensors were placed in small enclosures attached directly to the inlet and exit ports of the exposure chamber. The inlet detector measured the hydrogen peroxide concentration at the entrance to the enclosure. The exit detector measured the hydrogen peroxide concentration within the chamber immediately after the effluent exited the chamber. The sensors were factory preset to measure from 0 to 4000 ppmv H₂O₂, with sensitivity less than or equal to $\pm 5\%$ of the measured value. However, the sensors were recalibrated in-line using VHP concentration values determined by chemical titration of VHP captured in bubbler solutions. The inlet hydrogen peroxide detector was calibrated to measure from 0 to 800 ppmv H₂O₂, and the outlet hydrogen peroxide detector was calibrated to measure from 0 to 340 ppmv H₂O₂ IAW IOP DS04001. Sensor operation was verified during each run using the average value from three

iodometric titrations on the VHP stream entering and exiting the glove box (IOP DS04019). A Vaisala HUMICAP temperature and humidity sensor transmitter (model HMT333) was placed in the center of the chamber. The relative humidity sensor accuracy was $\pm 1\%$ at 0 to 90%RH, and $\pm 1.7\%$ at 90 to 100%RH (non-condensing). The temperature sensor operating range was -40 to 80°C , with an accuracy of $\pm 0.20^\circ\text{C}$. The sensor data was collected electronically using a portable data logging system manufactured by Omega Engineering (OMP-MODL). The data were transferred to a computer running the Omega-supplied, Microsoft Windows-based, HyperWare software for data plotting, real-time trending, and initial analysis. An Omega OMP-MLIM-4 expansion module was used to monitor output from the device. Data was collected at a rate of at least one data point per minute.

VHP was generated using a Steris VHP[®] M100-S hydrogen peroxide vapor-generating system and 35% hydrogen peroxide. Airflow and peroxide solution feed rate were controlled using a Siemens OP-7 interfacing unit. The desired flow rate and peroxide feed rate were entered into the interfacing unit. The VHP concentration ranges for this testing were 250 to 300 ppmv and 125 to 150 ppmv. The bottle containing hydrogen peroxide was not weighed before and after each test, therefore, the mass of decontaminant consumed was not recorded. The VHP decontamination technology requires relatively low humidity conditions to reduce the likelihood of peroxide condensation. The low humidity was maintained by drying the air with a Munters MG90 desiccant dehumidifier before it was fed into the VHP delivery system. The humidity of the air fed into the VHP delivery system was $<30\%$ RH at the start of test. The humidity of the air in the test chamber was typically $<15\%$ RH. Exposures were carried out at a minimum of 30°C . Temperature, humidity, and hydrogen peroxide concentrations were continuously monitored during the decontamination process. The offgas from the chamber was decomposed and released into a chemical fume hood. The VHP was catalytically decomposed to water and oxygen in a Random Technology catalytic converter, containing metal catalysts (platinum and palladium), on an aluminum honeycomb monolith substrate. The chamber was operated IAW SOP RNG-107, IOP DS04015, and IOP DS04016.¹⁴⁻¹⁶ A Drager Pac III single gas monitor, fitted with a Drager hydrogen peroxide sensor, was used to monitor the VHP outside the chamber in the workspace. The standard measuring range of the VHP monitor was 0 to 10.0 ppmv H_2O_2 , with a display resolution of 0.1 ppmv.

A small recirculation fan was used in the chamber to mimic the air circulation provided by fans in commercial large room decontamination. Air circulation was observed using a “fog” test of dry ice and warm water, rather than a “smoke” test. There was concern that the smoke test would leave residue inside the chamber that could interfere with the coupon studies.

4.3 Material Demand Testing

Each material type was tested independently, in three replicate trials, at both the target and half-target VHP concentrations. The number of test materials was dependent on the coupon surface area. The Material Demand test used the appropriate number of coupons so that the total surface area exposed to vapor was essentially the same for each coupon type. The sample surface area was calculated by summing the area for each exposed sample face (Table 2). For example, the wood surface area was $(2 \times 1 \times w) + (2 \times 1 \times h) + (2 \times w \times h)$. The testing was conducted in accordance with the procedures documented in SOP RNG-107 and IOPs DS04015 and DS04016 and as shown in Appendix B, Figure 24.¹⁴⁻¹⁶ The hydrogen peroxide sensor performance was verified before testing in accordance with IOP DS04015.

Table 2. Coupon exposed area.

Material	Code	Sample Dimensions			Sample Surface Area* (cm ²)	Coupons per Test	Total Area (cm ²)	Vapor per Sample Area** (cm ³ /cm ²)
		Length (cm)	Width (cm)	Height (cm)				
Structural Wood, Fir, Type-II	W	25.4	3.9	1.3	270	18	4863	65.2
Latex-Painted Gypsum Wallboard	G	15.2	15.2	1.3	539	9	4854	65.3
Concrete	C	10.2	20.3	1.4	495	10	4952	64.0
Carpet	R	15.0	20.0	-----	600	8	4800	66.1
Painted Structural Steel (Dog Bone Shape)	S	7.8	5.2	0.6	267	18	4798	66.1
		15.0	2.0	0.6				
Ceiling Suspension Tile, Acoustical	T	30.0	8.0	1.4	586	8	4691	67.6

* Sample surface area is calculated for each exposed sample surface.

**Volume chamber is 11.2 cubic feet (317,148 cubic cm).

4.3.1 Dhumidification

The coupons were exposed to decontaminant in accordance with Section 6.0 of the Material Demand QAPP.¹⁷ The coupons were placed in the exposure chamber in accordance with IOP DS04016.¹⁶ The chamber humidity was adjusted below 30%RH, using airflow from the dehumidifier, before VHP was introduced into the glove box. The time required to adjust the humidity in the chamber was between 15 to 30 min.

4.3.2 Conditioning

VHP was introduced into the chamber to reach the target concentration of 250 ppmv or the half-target concentration of 125 ppmv. Once the measured VHP concentration reached the target concentration, the decontamination phase was started.

4.3.3 Decontamination

The VHP concentration within the chamber was maintained within the target concentration range of 250 to 300 ppmv or the half-target concentration range of 125 to 150 ppmv. The same CT values (chamber concentration multiplied by time) were obtained for the target and half-target concentrations by running the half-target concentration tests for twice the time of the target concentration tests. During reliability tests with the VHP system, it was determined that the initial residence time of VHP in the chamber at 0.2 CFM (Requested by the EPA) was longer than the decomposition half-life. The concentration of VHP at the outlet detector was only 20% of the concentration measured at the inlet detector. After consultation with Steris, the chamber was fitted with larger diameter tubing to allow increased flow through the chamber and, therefore, a faster turnover rate. Further characterization/reliability tests (1 to 6 CFM) showed that increasing the flow through the chamber minimized the difference in the VHP concentrations at the inlet and outlet detectors. The flow rate (3.0 CFM) through the chamber was optimized for both residence time and decomposition of peroxide. The flow rate was documented in the Quality Assurance Project and Work Plan (QAPP).⁷ The flow rate provided a turnover of approximately 16 exchanges per hour in the chamber. The flow rate was fixed at 3 CFM for each stage of the Material Demand tests (dehumidification, conditioning, decontamination, and aeration). The temperature during exposure

was kept above the minimum requirement of 30 °C. The hydrogen peroxide sensor performance was verified at least once during each test by comparison to a wet-chemical titration method.

4.3.4 Aeration

Aeration of the chamber was conducted following the decontamination period. The VHP concentration within the chamber was monitored until end-of-run (EOR). EOR was defined as the reduction of chamber concentration to 10% of the decontamination concentration. For the VHP studies, EOR was approximately 15 ppmv for half-target or 30 ppmv for target concentration runs. Aeration of the chamber continued until the vapor concentration was equal to or below the levels required by the Risk Reduction Office to assure safe operation for personnel. The procedures for the safe opening of the chamber and coupon removal after fumigant exposure are documented in SOP RNG-107 and IOP DS04015. Low-level vapor monitors were used for monitoring personnel. A Dräger Pac III single gas monitor, fitted with a Dräger hydrogen peroxide sensor, was used to monitor the VHP. The standard measuring range of the VHP monitor was 0 to 10.0 ppmv H₂O₂, with a display resolution of 0.1 ppmv.

4.4 Data Review and Technical Systems Audits

The approved Material Demand QAPP specified procedures for the review of data and independent technical system audits. All test data were peer reviewed within two weeks of generation. The project quality manager (or designee) was required to audit at least 10% of the data. In addition, the project quality manager (or designee) performed four technical system audits over the course of testing. A technical system audit is a thorough, systematic, on-site qualitative audit of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of the system. The QA findings are documented in Section 8.

4.5 Material Demand Calculation

The difference between the target chamber CT (CT_{outlet,t} in ppmv-hr) and the inlet CT (CT_{inlet,t} ppmv-hr) required to achieve the target (1000 ppmv-hr) can be attributed to the demand of the material in the chamber for VHP. This demand is comprised of reversible adsorption (e.g., physisorption) and chemical reaction (e.g., decomposition or chemisorption) on the surfaces within the chamber. A contribution of homogeneous decomposition may also be present; however, efforts were made to minimize the contribution of this mechanism (e.g., rapid turnover rate and shielding from UV). A correction must be made for the hydrogen peroxide remaining in the chamber at the end of the fumigation period. This correction factor, CT_{charge}, was determined by multiplying the volume of the chamber by the concentration in the chamber at the end of the fumigation period and converting it into terms of ppm-hrs using the following equations.

The impact of each material on the required CT (CT_{diff,k}) can be determined by subtracting the observed difference in CT in the baseline tests (CT_{diff,b}) from that observed with a specific material type in the chamber (CT_{diff,mb}). This is shown in Equation 1. It is important to note that the CT is generally not calculated until the target concentration has been reached during fumigation in the field. However, for the purposes of this research, the CT begins at the time injection is started to account for any sorption occurring before the target concentration is reached.

$$CT_{diff,k} = CT_{diff,mb} - CT_{diff,b} = (CT_{outlet} - CT_{inlet} - CT_{charge})_{mb} - (CT_{outlet} - CT_{inlet} - CT_{charge})_b \quad \text{Equation 1}$$

The time and surface-area-specific material demand (MD) over the fumigation period (up to 1000 ppmv-hr) can be calculated according to Equation 2, where CT_{diff,k} is divided by the

material surface area (A , in m^2) and the time required to reach the target CT_{outlet} (t , in hr). The units of MD are $ppmv\cdot h/m^2$. The total surface area added to the chamber for each material type is reported in Table 2. The total interior surface area of the chamber and material support structures was $3.8 m^2$.

$$MD_k = -\frac{CT_{diff,k}}{tA} \text{ (for materials), } MD_b = -\frac{CT_{diff,b}}{tA} \text{ (for baseline)} \quad \text{Equation 2}$$

The calculation of the material demand via Equation 2 determines the relative effect of each material on the chamber VHP concentration.

The Material Demand is also reported in $g/m^2/h$ for the chamber (Table 3) and the materials (Table 4 and Table 5). The mass of VHP decomposed or sorbed by a specific material, $M_{H_2O_2,k}$ (g), was calculated from the $CT_{diff,mb}$ and $CT_{diff,b}$ by Equation 3.

$$M_{H_2O_2} = \frac{CT_{ppmv\cdot hr} MW_{H_2O_2} P_{sys}}{1000RT_{sys}} F_{total} \quad \text{Equation 3}$$

where:

$MW_{H_2O_2}$	=	the molecular weight of hydrogen peroxide, 34 g/gmole
P_{sys}	=	the chamber pressure in atmospheres
T_{sys}	=	the chamber temperature in K
F_{total}	=	the flowrate through the chamber, 5097 L/h
R	=	the universal gas constant (0.0826 L atm/gmole K)

The mass flux for the baseline tests, J_b ($g/m^2/h$) was then calculated using Equation 4.

$$J_b = M_{H_2O_2,b} / tA \quad \text{Equation 4}$$

The mass flux for each material, J_k ($g/m^2/h$), was calculated using Equation 5.

$$J_k = (M_{H_2O_2,mb} - M_{H_2O_2,b}) / tA \quad \text{Equation 5}$$

The baseline correction was required because $CT_{diff,mb}$ included homogeneous decomposition, and due to the material demand caused by the interior of the exposure chamber.

The average and standard deviation of each specific test were calculated using Microsoft® Office Excel (2003) SP2 software. The determination of statistical outliers was performed according to the Grubb's test, also known as the extreme studentized deviate (ESD) method. No data was discarded as an outlier within a data set (i.e., set of triplicate experiments at each concentration for each material).

5. EVALUATION OF EMPTY CHAMBER

5.1 "Fog" Test Results and Discussion

A "Fog" test was conducted to observe the chamber air circulation pattern created by the chamber recirculation fan. The test was conducted with an airflow of 3.0 CFM through the chamber. The small recirculation fan was used in the chamber to mimic the air circulation provided

by fans in the commercial decontamination of a large room. The fan was placed on the bottom of the chamber in the back right corner. The fan blew toward the opposite corner of the chamber. The “Fog” test was used to verify that the coupons placed on the exposure rack would have decontaminant vapor contact during testing. A container of dry ice and warm water was placed in the chamber. The fog produced could be sustained for several minutes. Air was introduced into the chamber on the lower right side of the chamber and the flow was observed. Figure 4 shows the photographs taken of the fog test within the exposure chamber. The density of the fog was hard to photograph; however, the fog developed an even density and did not stratify.

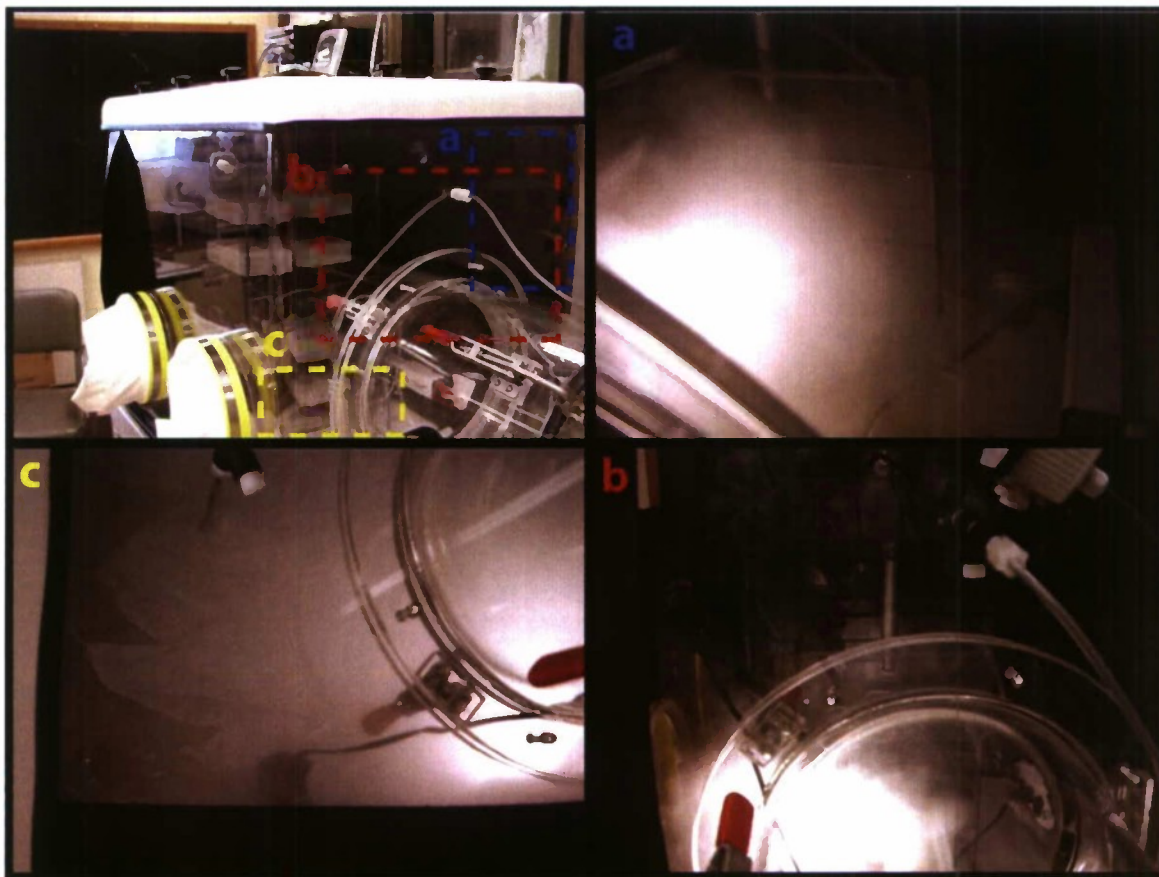


Figure 4. Exposure chamber fog test.

5.2 Baseline Tests and Results

Three baseline tests, for the determination of VHP loss due to spontaneous decomposition and/or adsorption and decomposition from the chamber interior surfaces, were conducted at the target and half-target concentrations. The results on those tests are presented in Table 3. No coupons were used in the baseline tests. The sample rack was in the chamber during the baseline tests. The airflow rate during the baseline tests was 3.0 CFM.

The hydrogen peroxide sensor performance was verified using a wet-chemical titration procedure during testing. For the first three test runs, the titration was performed at the beginning, middle, and end of the run. After the first three tests, the titration was performed during the

first half of the run. Three replicate samples were collected and titrated. The average agreement between the inlet sensor reading for the hydrogen peroxide and the titration results for the nine titration tests, performed for the target and half-target VHP runs, were 4.2 and 4.8%, respectively. The average agreement between the outlet sensor reading for the hydrogen peroxide and the titration results for the nine titration tests, performed for the target and half-target VHP runs, were 3.5 and 3.8%, respectively.

Zero time on all graphs signifies the time when the VHP concentration within the enclosure first reached the minimum value of the concentration range—either 250 or 125 ppmv (Figure 5). Based on the four-step VHP process, zero time was the start of the decontamination phase.

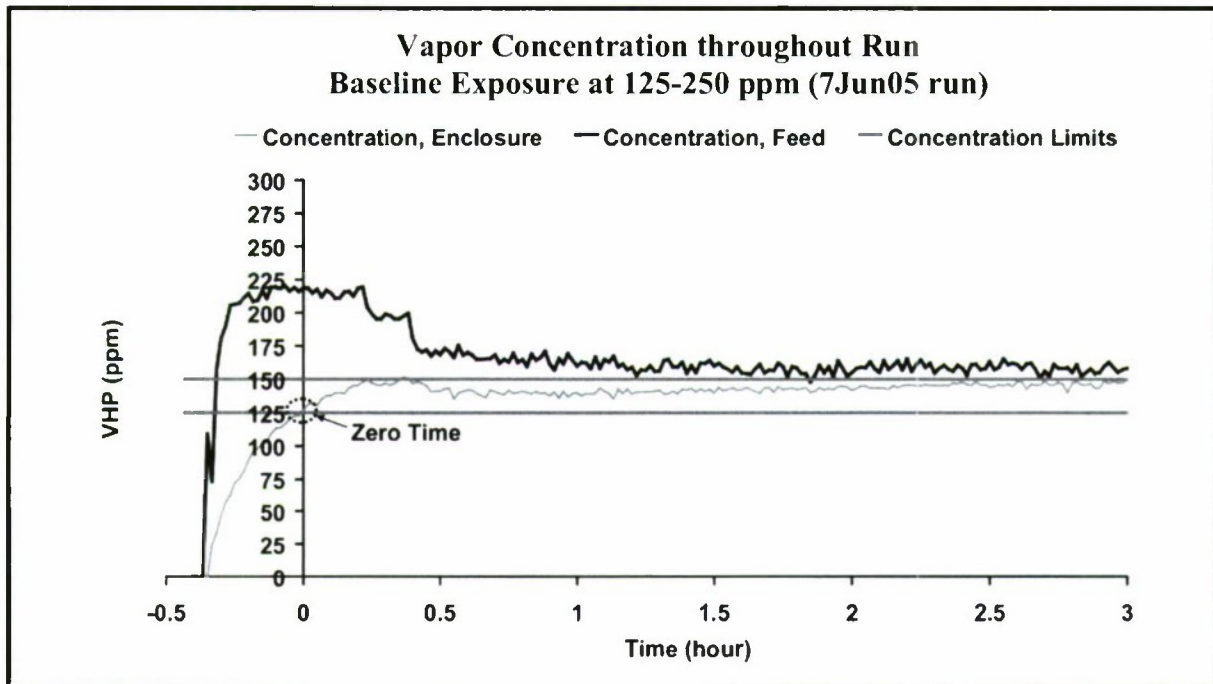


Figure 5. Illustration of the determination of zero time.

Table 3. Baseline material demand test results.

Test	Ave. Chamber Temp. (°C)	Ave. Feed Conc. (ppm)	Time to reach target CT (hr)	ΔCT (ppmv-hr)	MD (ppmv-hr/hr/m ²)	J (g/hr/m ²)
Baseline (125-200 ppmv)	32.5 ± 1.3	151.2 ± 3.2	7.47 ± 0.16	128.3 ± 4.6	4.18 ± 0.16	0.0272 ± 0.0014
Baseline (250-300 ppmv)	32.8 ± 0.1	326.4 ± 6.7	3.73 ± 0.08	199.2 ± 9.8	13.90 ± 0.66	0.0896 ± 0.0046

Representative VHP 125 and 250 ppmv baseline test CT graphs are shown in Figure 6. The CT within the chamber and the CT of the feed air are shown in gray and black, respectively. The feed concentration was adjusted as needed to maintain the chamber concentration within the target concentration range (Figure 7).

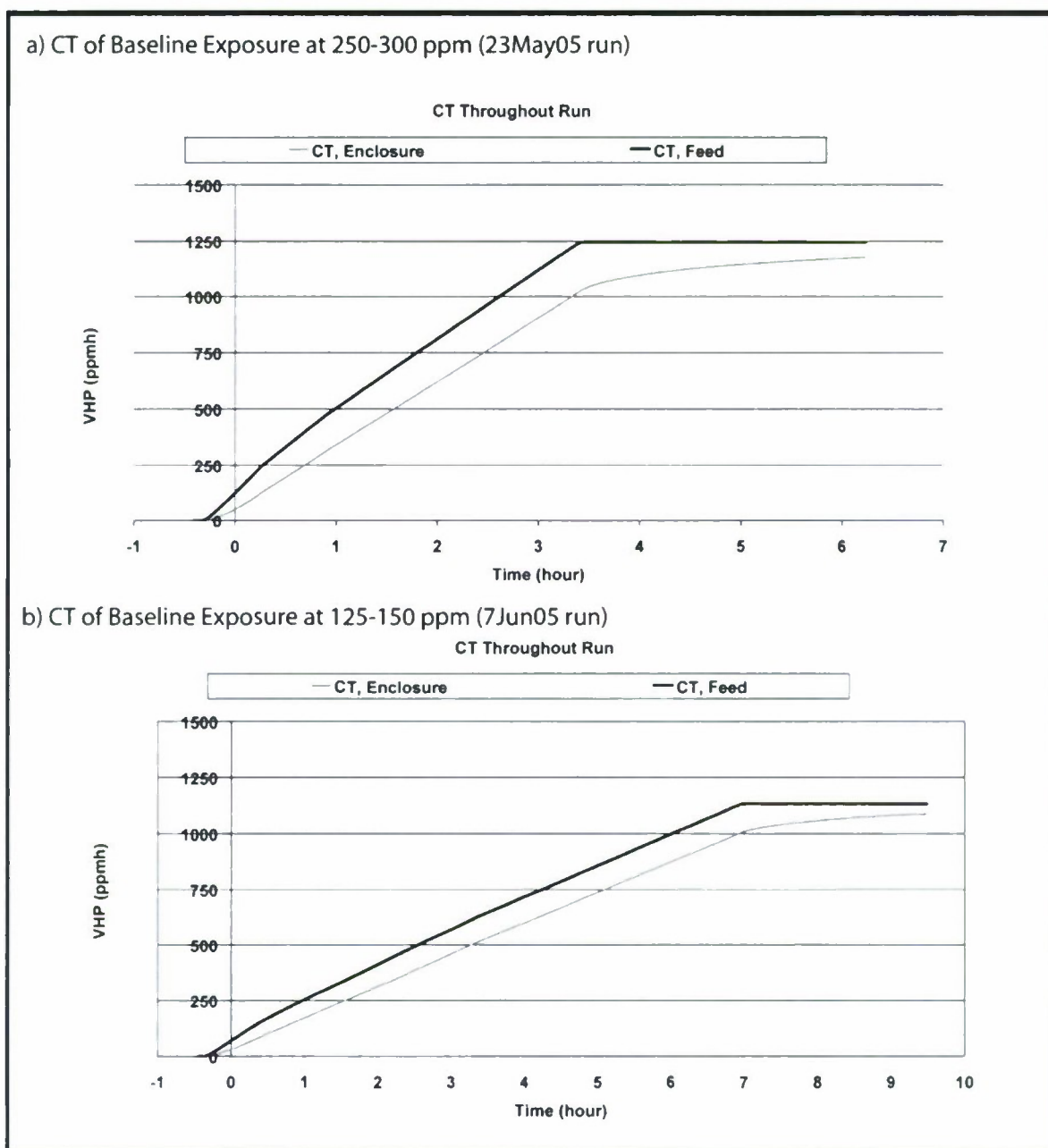
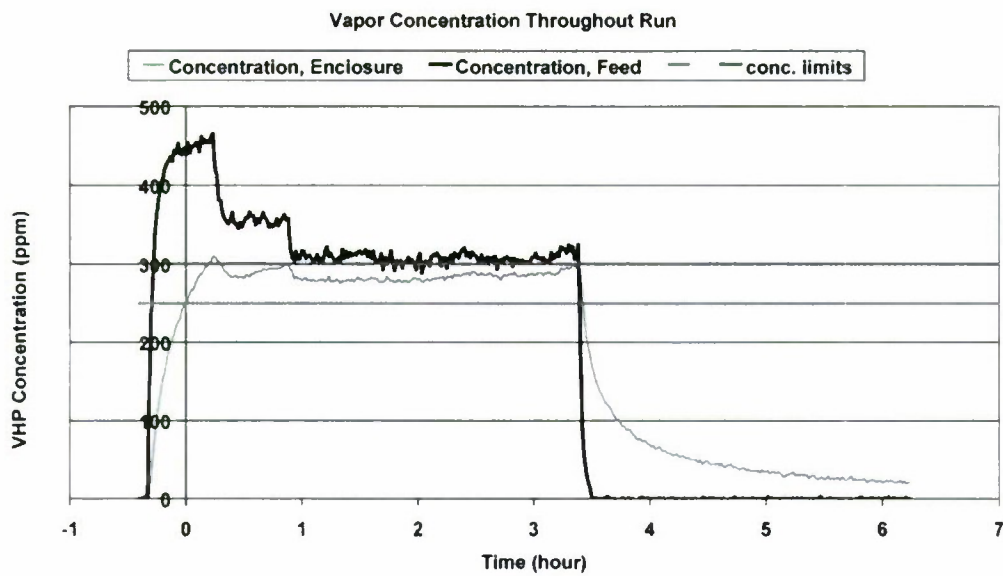


Figure 6. Baseline VHP exposure tests.

a) Concentration Profile for Baseline Exposure at 250-300 ppm (23May05 run)



b) Concentration Profile for Baseline Exposure at 125-150 ppm (7Jun05 run)

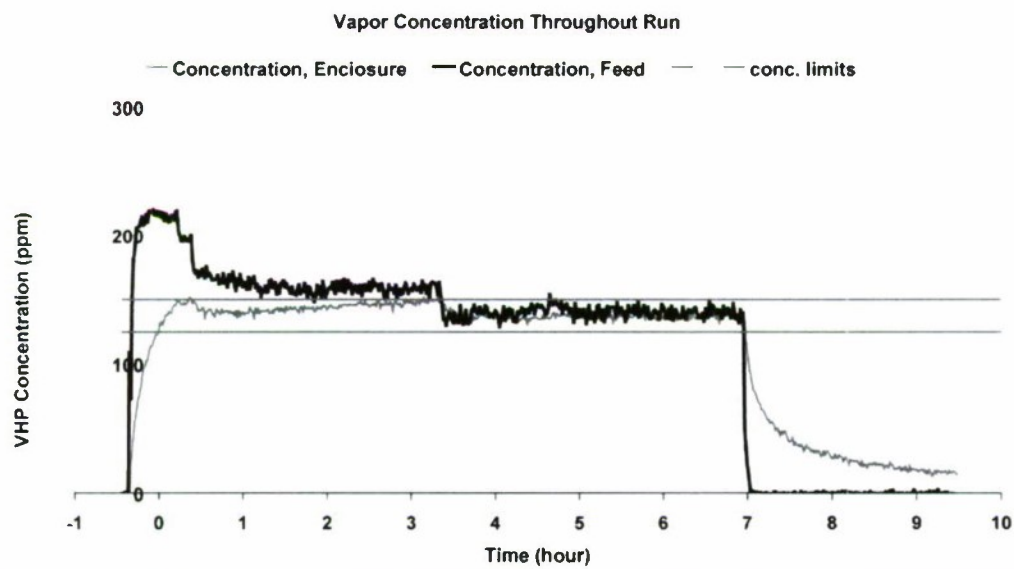


Figure 7. Concentration profile for baseline VHP tests.

5.3 Baseline Test Discussion

The baseline test results showed minimal hydrogen peroxide loss due to spontaneous decomposition and/or adsorption and decomposition from the chamber interior surfaces at 3 CFM. The hydrogen peroxide sensor performance showed good agreement with the wet-chemical titration results.

The dehumidification step was conducted prior to the start of data collection. Data collection began with the introduction of fumigant into the chamber during the conditioning phase. Once the VHP concentration reached the target concentration, the feed rate was reduced and the decontamination phase began. In the field, the cumulative CT calculation would not begin until this point; however, in this study, the CT was calculated at the start of hydrogen peroxide injection into the chamber, resulting in an exposure less than what would be experienced in the field. An attempt to correct this discrepancy, by extrapolating the data out to a true CT of 1000 ppmv-h, showed that the difference was not statistical. Because the difference was insignificant, the results reported in this report were based on starting the CT calculation at the start of injection. The feed concentration was reduced to zero once the target CT had been reached by immediately stopping the liquid peroxide injection into the VHP generator. Once the liquid peroxide injection was terminated, the decontamination phase ended and the aeration phase began (Figure 8). The immediate termination of liquid hydrogen peroxide injection resulted in the sharp flattening of the feed CT curve (Figure 6). The enclosure CT did not immediately flatten out. The enclosure CT continued to rise at a slower rate, after reaching 1000 ppmv · h, due to flow rate and chamber volume. The enclosure hydrogen peroxide concentration decreased as the VHP was diluted with the input air.

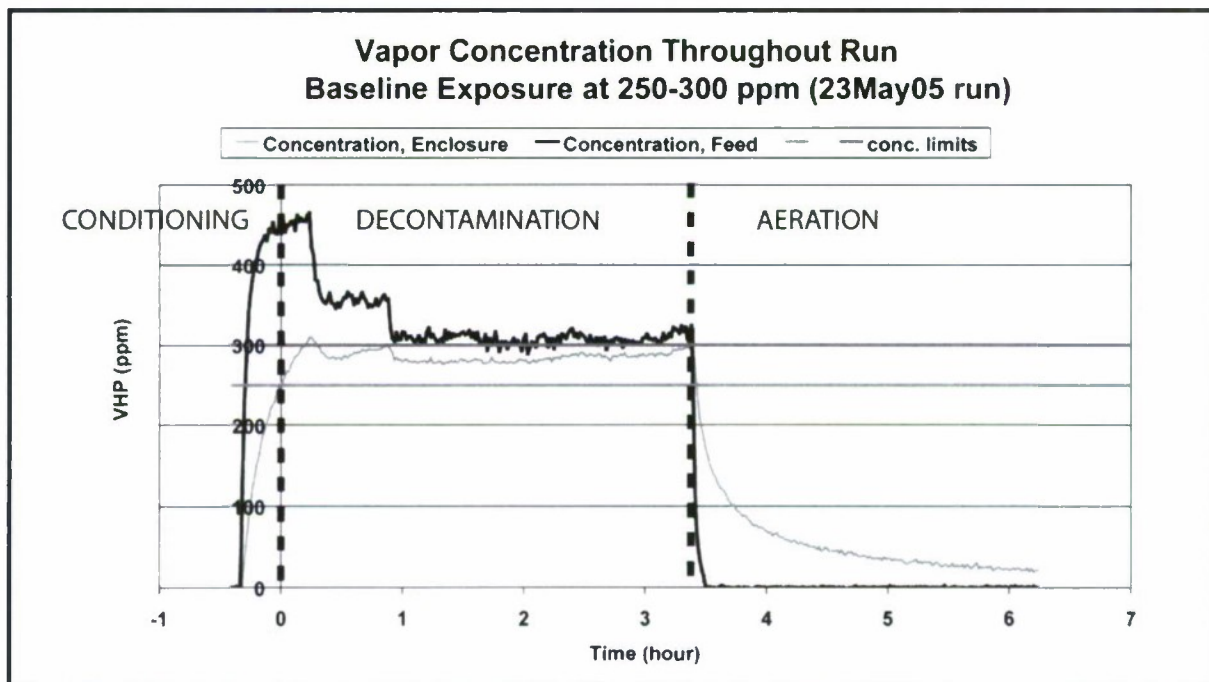


Figure 8. VHP process using a baseline test.

Data for CT curves were collected until the VHP concentrations within the chambers dropped to $\leq 10\%$ of the decontamination concentration. The difference between the feed CT and

enclosure CT curves was due to loss of VHP within the enclosure. Loss of VHP during the baseline test could have resulted from spontaneous VHP decomposition, VHP decomposition on chamber surfaces, and/or surface sorption. To minimize potential loss due to condensation, the chamber, sensor enclosures, and tubing were wrapped with insulation and maintained at temperatures above 30 °C. Similarly, potential losses due to light exposure can be disregarded because the chamber, sensor enclosures, and tubing were either opaque or wrapped with opaque insulation.

6. EVALUATION OF BUILDING MATERIALS

6.1 Results

The VHP Material Demand chamber exposure tests were conducted from February through June 2005. The exposure chamber temperature profile was maintained within a small range of 30 to 35 °C throughout testing (Figure 9).

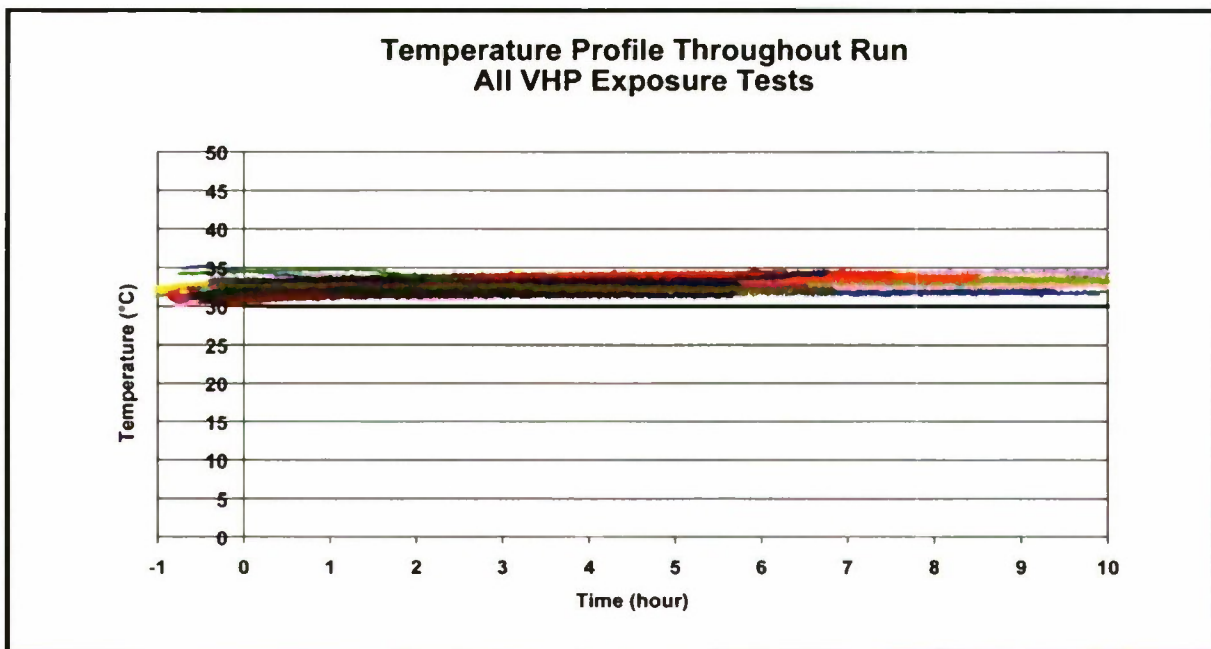


Figure 9. Chamber temperature profile throughout testing.

In the presence of VHP, at approximately 300 ppmv, the Vaisala HUMICAP humidity sensor (model HMT333) reading was slightly high. But when removed from air containing VHP, the sensors responded normally with no change in response. Because the relative humidity constraint was applicable to the condition of the chamber before the fumigant was introduced, the condition was satisfactorily met with the sensors. Additional evaluation of the relative humidity sensors is provided in Section 8.1.

The hydrogen peroxide sensor performance was also verified for each material exposure run using the wet-chemical titration procedure. The titration was performed during the first half of each run. Three replicate samples were collected and titrated. The average agreement between the inlet sensor reading for the hydrogen peroxide and the titration results for the 18 titration tests, performed for the target and half-target VHP runs, were 6.5 and 3.2%, respectively. The average

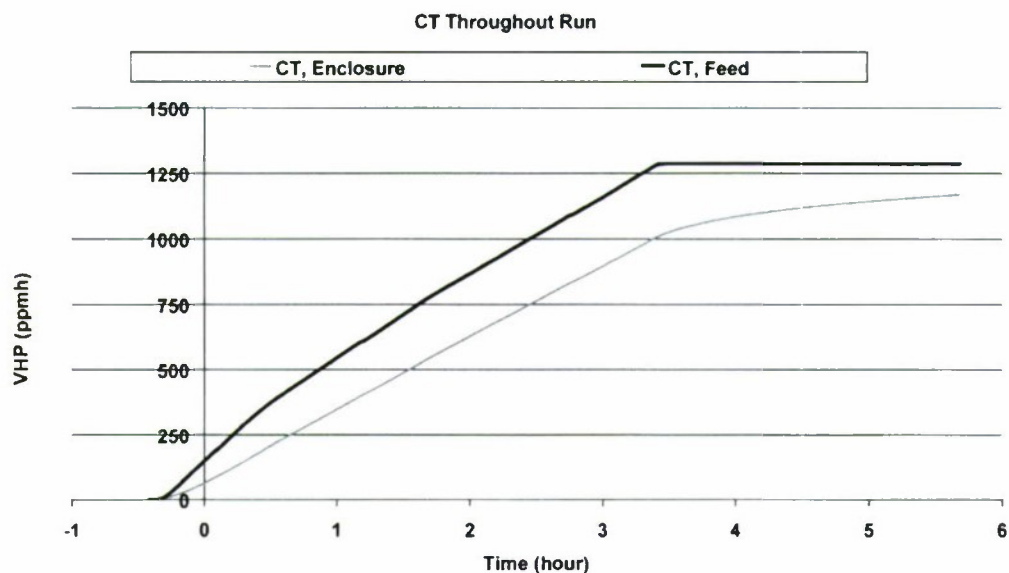
agreement between the outlet sensor reading for the hydrogen peroxide and the titration results for the 18 titration tests, performed for the target and half-target VHP runs, were 5.4 and 4.2%, respectively. The performance verification of the hydrogen peroxide sensor, conducted during each run, showed that neither sensor was adversely affected by prolonged and repeated exposure to VHP. Neither of the two sensors showed evidence of visual deterioration or change in response during the testing period.

Representative CT graphs for each test material are shown in Figure 10 through Figure 15. Each graph consists of two CT curves. The enclosure CT (gray line) reflects the hydrogen peroxide CT within the chamber. The enclosure CT determined the test run duration. The concentration in the enclosure was maintained during the decontamination phase within either the target or half-target concentration range. The feed CT (black line) shows the hydrogen peroxide CT from the generator. The generator feed concentration was adjusted to maintain the chamber within the target or half-target concentration range during the decontamination phase.

The Material Demand contributions attributable to each of the building materials are shown in Table 4 and Table 5. The materials are listed in order of their ability to decrease the VHP concentration in the 125 to 150 ppmv exposures: carpet, painted structural steel, latex-painted wallboard, ceiling tile, wood, and concrete cinder block. The order is similar for the 250 to 300 ppmv test exposures with the exception of ceiling tile and wood, which were reversed for these exposures. The values shown are the average of three replicate exposures. Of the materials studied under both concentrations, concrete cinder block had the greatest effect.

Representative concentration profile graphs for each test material are shown in Figure 16 through Figure 21. Each graph consists of two concentration profiles. The enclosure concentration (gray line) reflects the hydrogen peroxide concentration within the chamber during the test's duration. During the decontamination phase, the concentration of the enclosure was maintained within either the target or half-target concentration range. The feed concentration (black line) shows the hydrogen peroxide concentration exiting the generator. During the decontamination phase, the generator feed concentration was adjusted to maintain the chamber within the target or half-target concentration range. Figure 19a shows small dips in the measured concentration. The occasional dip was caused by a small air bubble drawn into the vaporizer feed tube from the hydrogen peroxide solution.

a) CT of Carpet VHP Exposure at 250-300 ppm (2May05 run)



b) CT of Carpet VHP Exposure at 125-150 ppm (20May05 run)

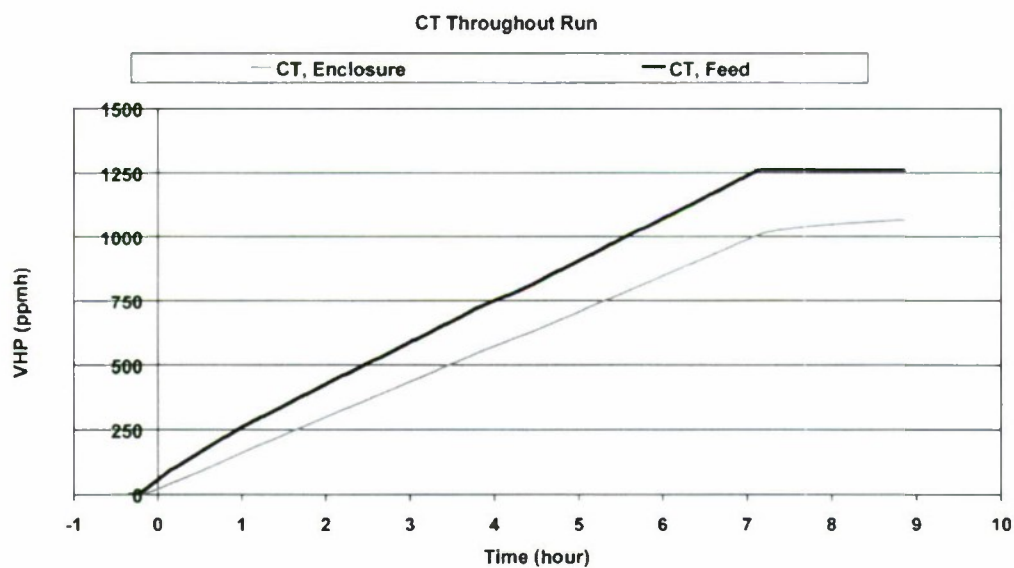
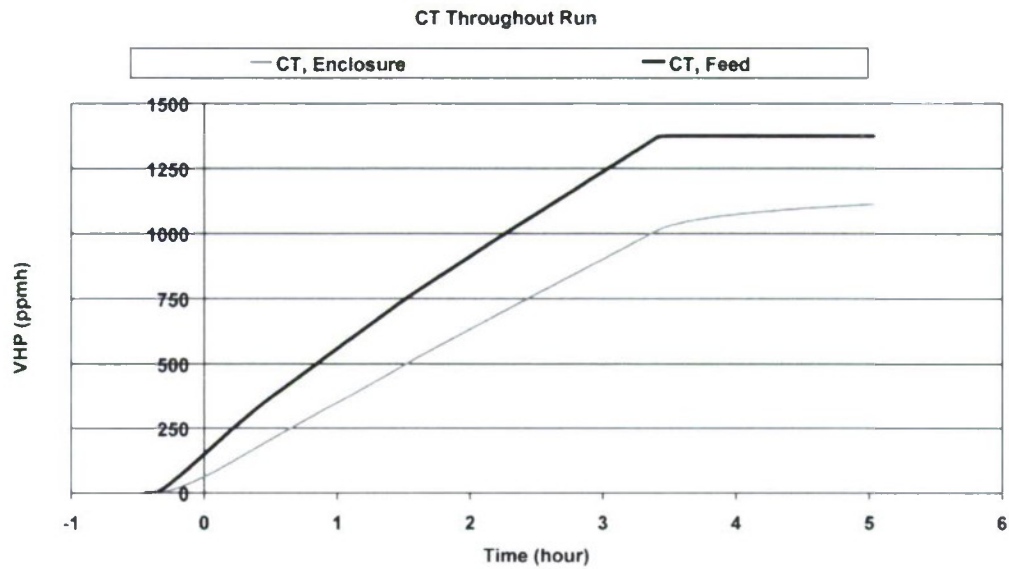


Figure 10. Representative VHP test results for carpet.

a) CT of Steel VHP Exposure at 250-300 ppm (18May05 run)



b) CT of Steel VHP Exposure at 125-150 ppm (6Jun05 run)

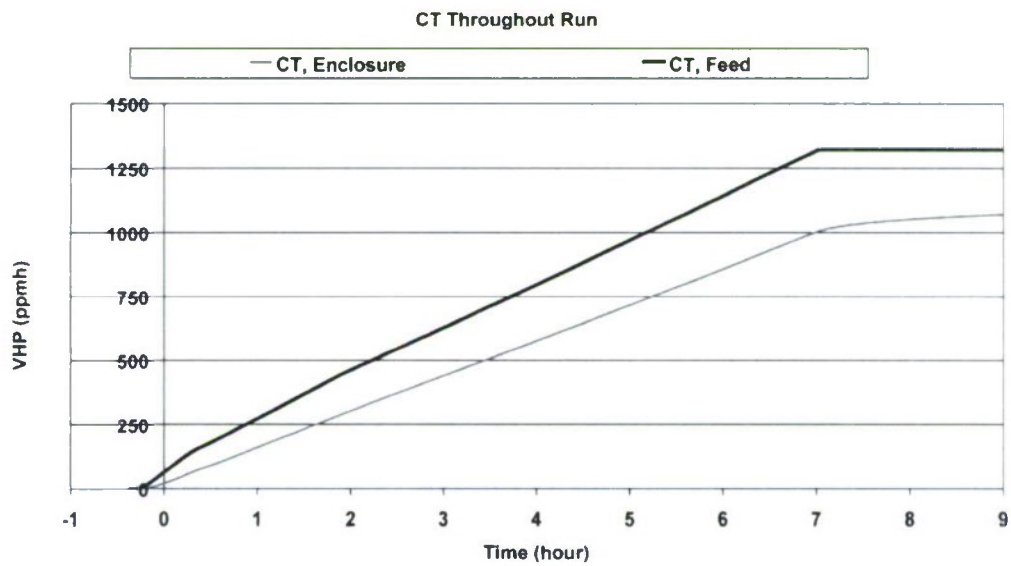
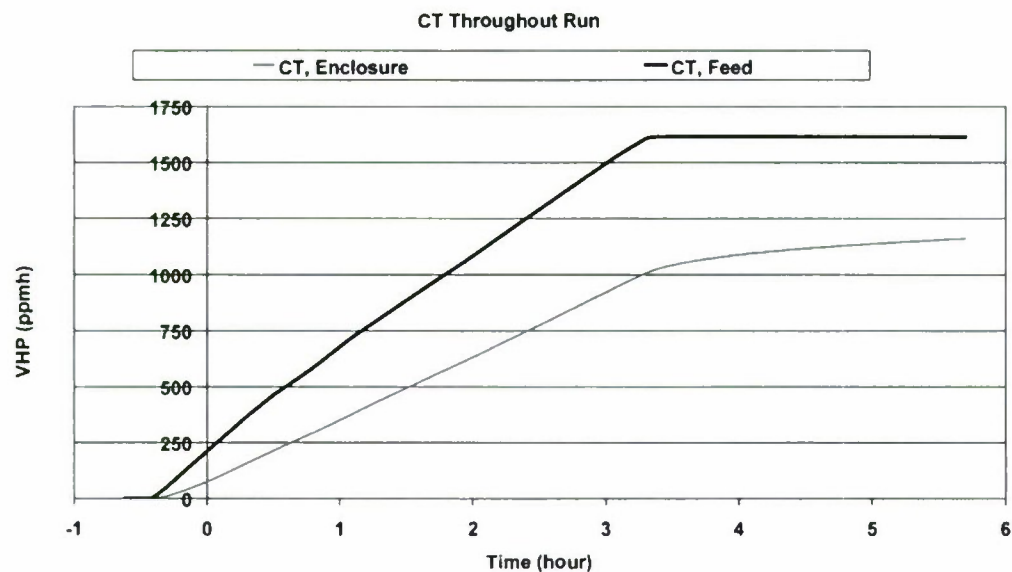


Figure 11. Representative VHP test results for steel.

a) CT of Gypsum Wallboard Exposure at 250-300 ppm (6May05 run)



b) CT of Gypsum Wallboard VHP Exposure at 125-150 ppm (31May05 run)

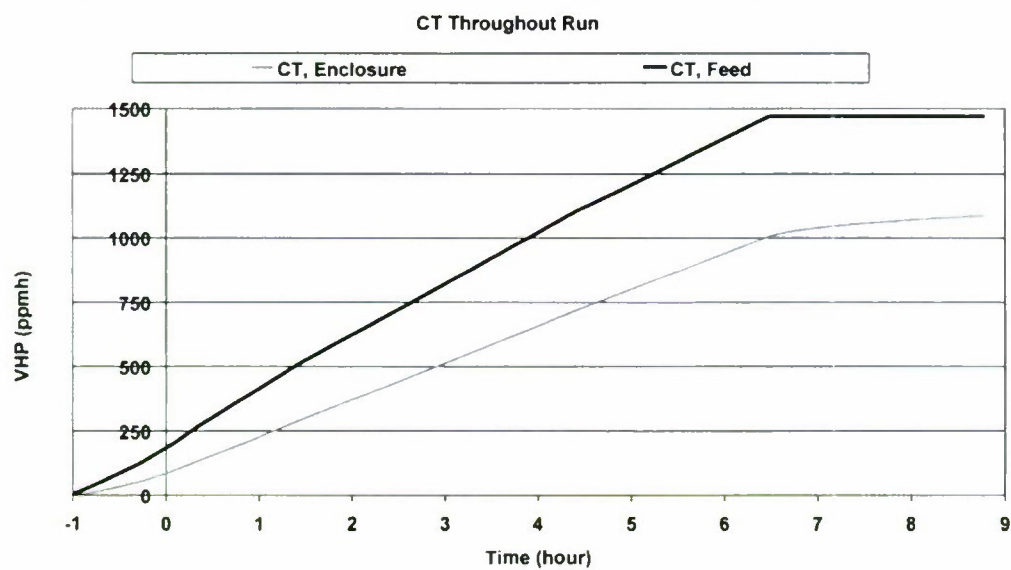
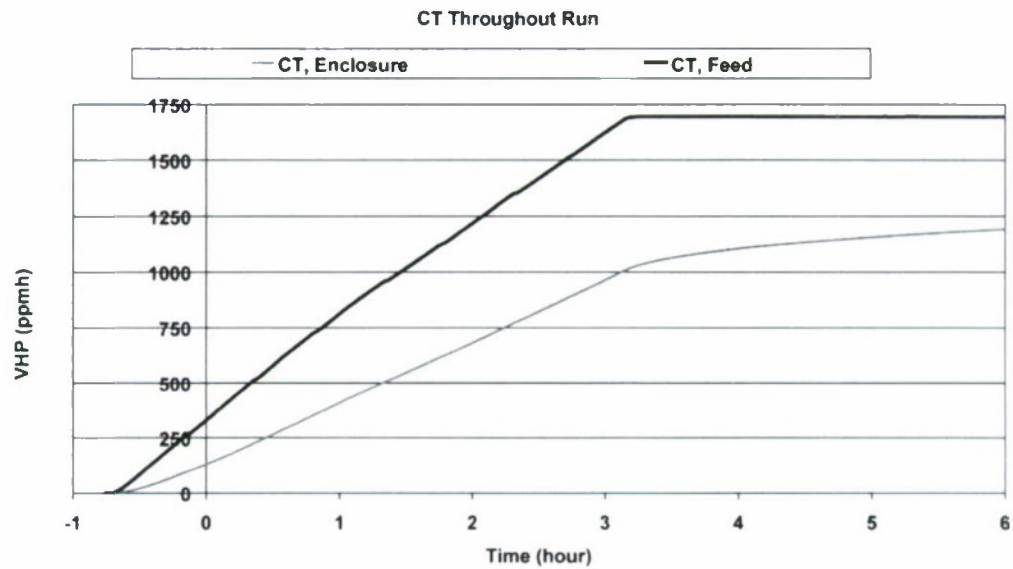


Figure 12. Representative VHP test results for painted gypsum wallboard.

a) CT of Acoustical Ceiling Tile VHP Exposure at 250-300 ppm (28Apr05 run)



b) CT of Acoustical Ceiling Tile VHP Exposure at 125-150 ppm (1Jun05 run)

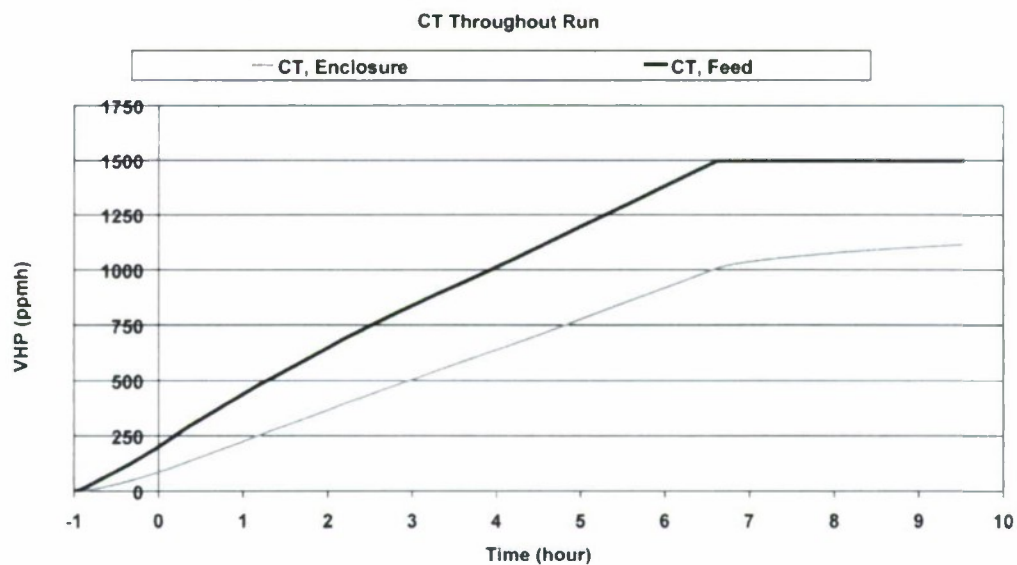
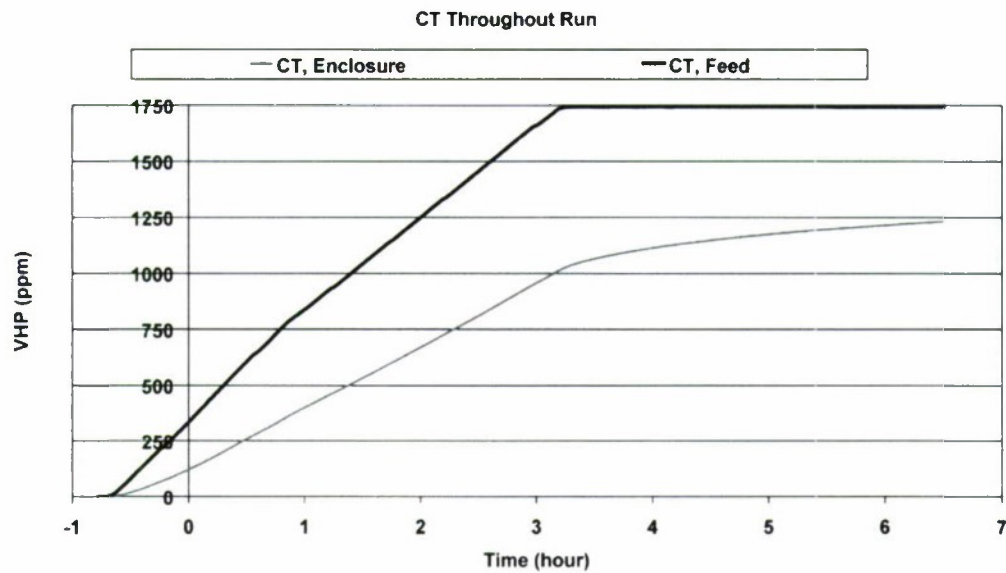


Figure 13. Representative VHP test results for acoustical ceiling tile.

a) CT of Wood VHP Exposure at 250-300 ppm (26Apr05 run)



b) CT of Wood VHP Exposure at 125-150 ppm (20May05 run)

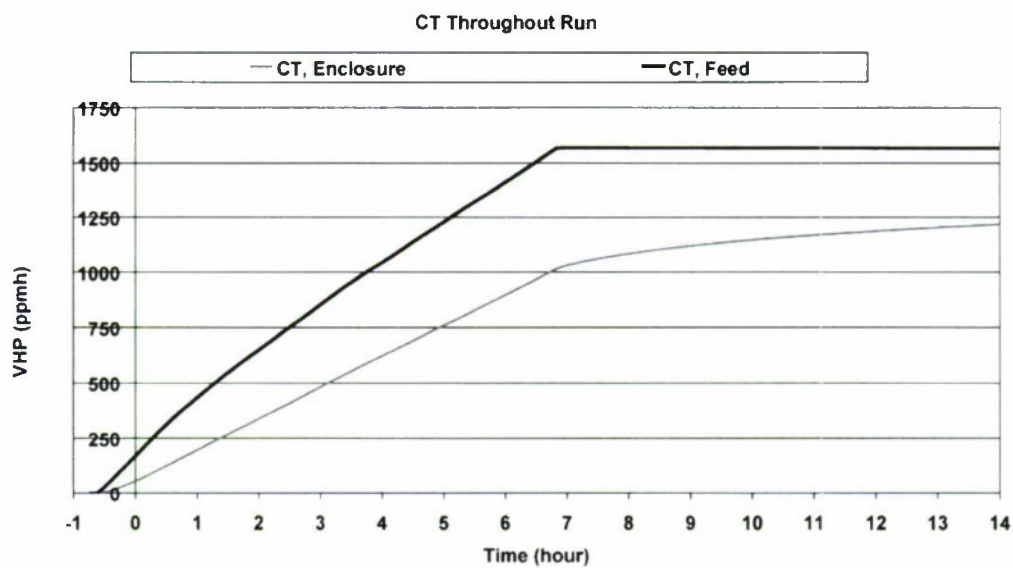
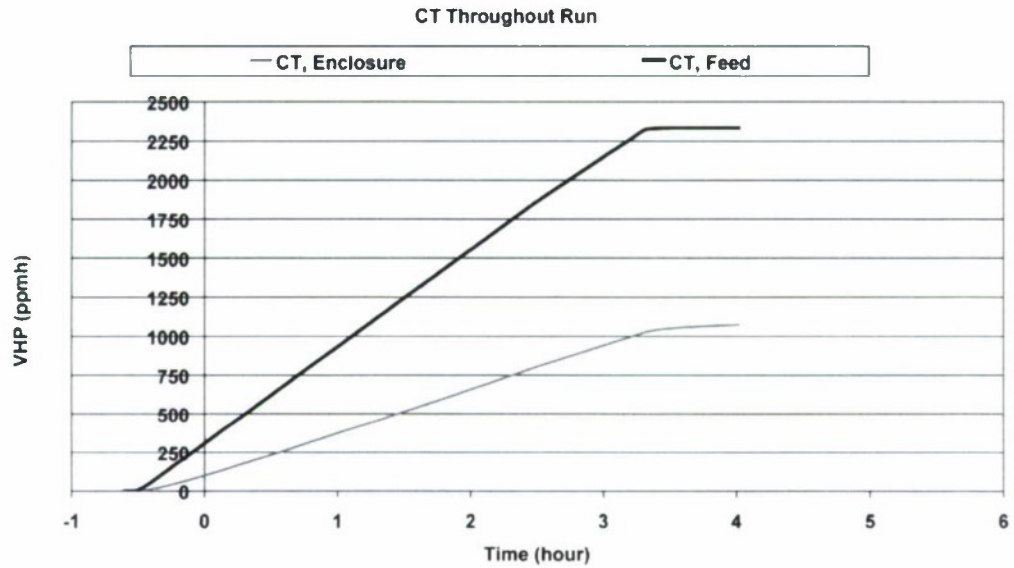


Figure 14. Representative VHP test results for wood.

a) CT of Concrete Cinder Block VHP Exposure at 250-300 ppm (4May05 run)



b) CT of Concrete Cinder Block VHP Exposure at 125-150 ppm (2Jun05 run)

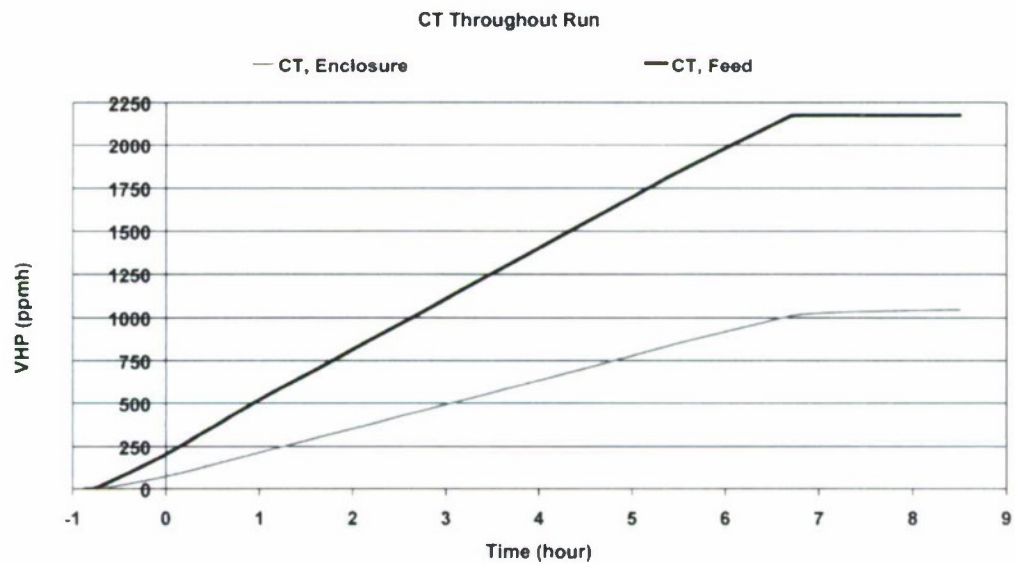


Figure 15. Representative VHP test results for concrete cinder block.

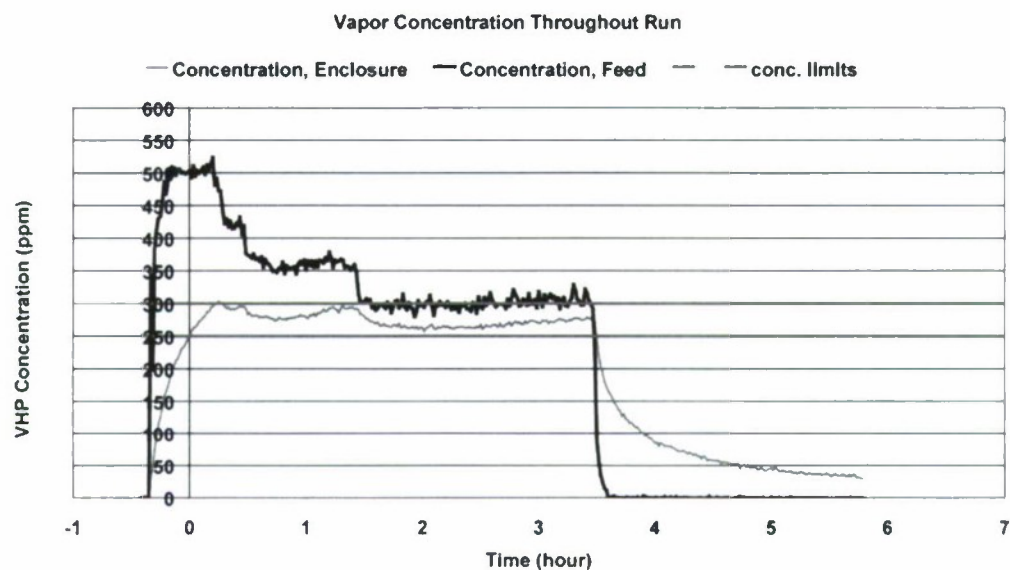
Table 4. Material demand results for building materials (125–150 ppmv).

Test	Average Chamber Temp. (°C)	Average Feed Conc. (ppm)	Time to reach target CT (hr)	Δ CT (ppmv-hr)	MD (ppmv-hr/hr/m ²)	J (g/hr/m ²)
Carpet	33.0 ± 0.7	163.4 ± 3.2	7.29 ± 0.14	62.4 ± 6.0	17.83 ± 1.40	0.111 ± 0.016
Steel	33.2 ± 0.5	174.4 ± 6.4	7.36 ± 0.13	154.8 ± 31.3	43.89 ± 9.35	0.274 ± 0.054
Wallboard	33.1 ± 0.1	197.5 ± 2.6	7.42 ± 0.07	336.0 ± 14.5	93.33 ± 4.06	0.591 ± 0.024
Ceiling Tile	33.1 ± 1.1	198.7 ± 4.6	7.51 ± 0.22	363.2 ± 14.5	103.07 ± 2.41	0.653 ± 0.041
Wood	32.8 ± 0.3	206.1 ± 7.1	7.41 ± 0.04	398.9 ± 49.8	110.69 ± 13.97	0.709 ± 0.083
Concrete	33.5 ± 0.2	299.2 ± 9.8	7.43 ± 0.03	1095.6 ± 82.7	297.58 ± 21.13	1.870 ± 0.123

Table 5. Material demand results for building materials (250–300 ppmv).

Test	Average Chamber Temp. (°C)	Average Feed Conc. (ppm)	Time to reach target CT (hr)	Δ CT (ppmv-hr)	MD (ppmv-hr/hr/m ²)	J (g/hr/m ²)
Carpet	31.9 ± 0.7	344.0 ± 5.1	3.79 ± 0.03	87.6 ± 20.8	48.15 ± 11.34	0.338 ± 0.073
Steel	33.1 ± 0.6	358.1 ± 3.3	3.76 ± 0.03	130.8 ± 21.2	72.44 ± 11.33	0.455 ± 0.050
Wallboard	32.2 ± 0.4	429.1 ± 3.9	3.73 ± 0.01	383.7 ± 18.7	212.03 ± 9.86	1.403 ± 0.075
Ceiling Tile	33.2 ± 0.8	436.4 ± 3.8	3.82 ± 0.02	451.3 ± 7.5	251.69 ± 4.87	1.591 ± 0.062
Wood	32.3 ± 0.2	439.9 ± 7.9	3.83 ± 0.04	466.9 ± 48.1	250.64 ± 23.28	1.650 ± 0.162
Concrete	32.2 ± 0.3	594.5 ± 10.2	3.77 ± 0.06	1022.9 ± 46.1	548.32 ± 21.07	3.612 ± 0.110

a) Concentration Profile for Carpet, VHP Exposure at 250-300 ppm (29Apr05 run)



b) Concentration Profile for of Carpet, VHP Exposure at 125-150 ppm (20May05 run)

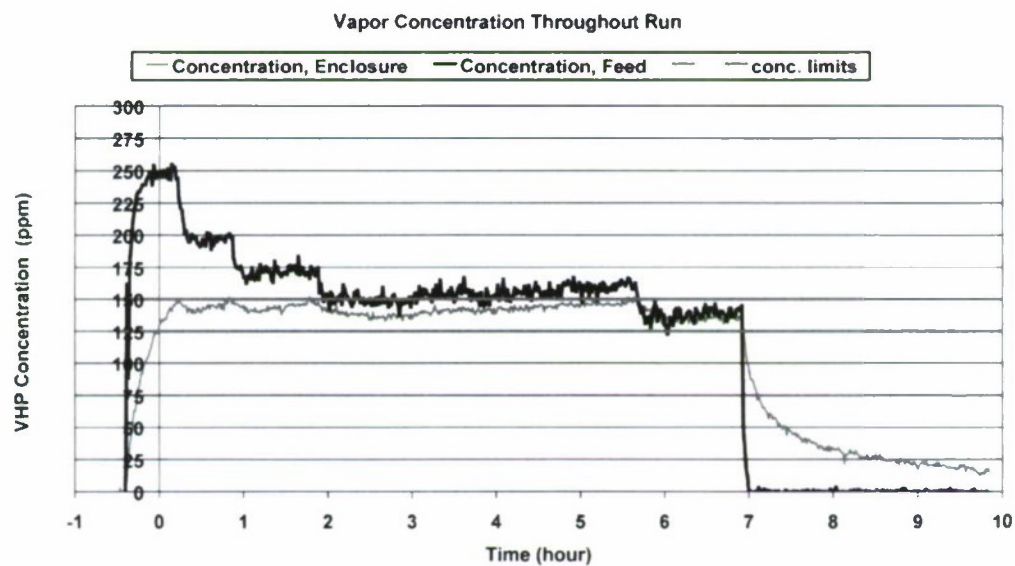
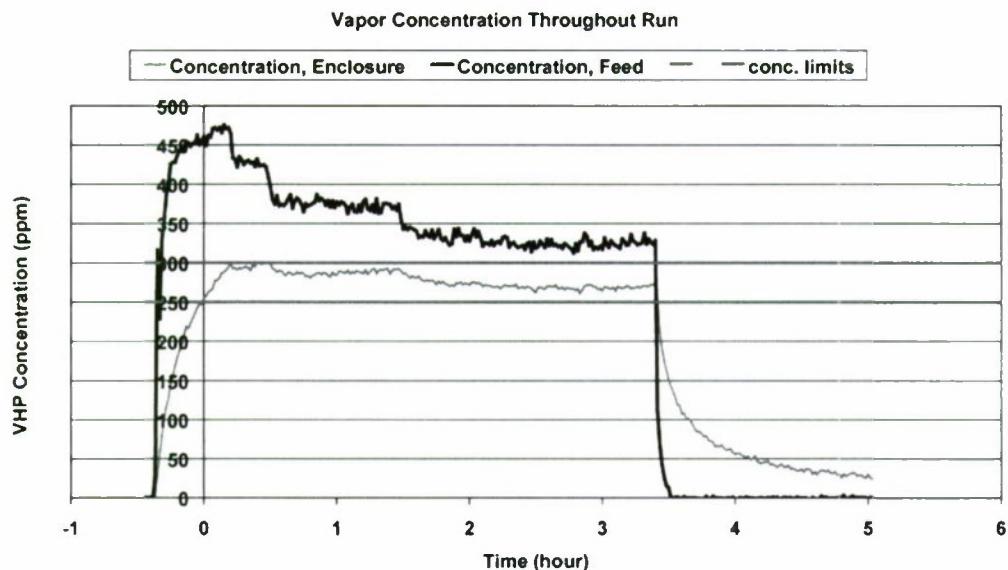


Figure 16. Representative concentration profile results for carpet.

a) Concentration Profile for Steel, VHP Exposure at 250-300 ppm (18May05 run)



b) Concentration Profile for Steel, VHP Exposure at 125-150 ppm (6Jun05 run)

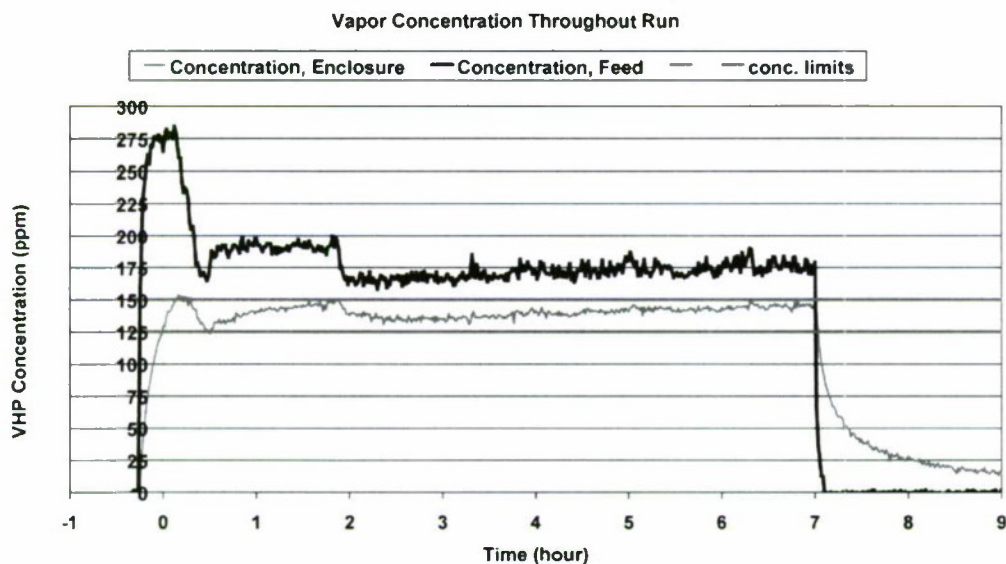
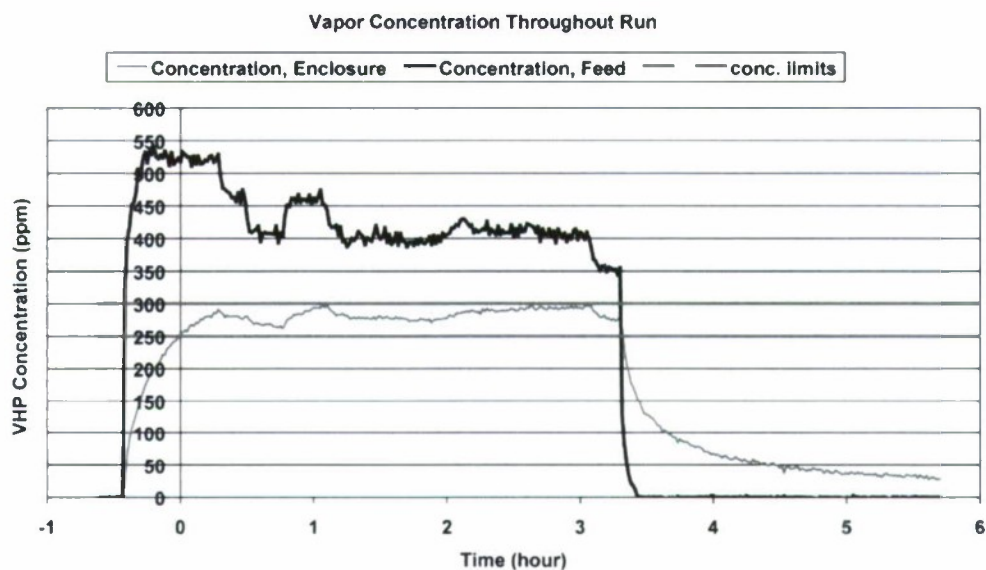


Figure 17. Representative concentration profile results for steel.

a) Concentration Profile for Gypsum Wallboard, Exposure at 250-300 ppm (6May05 run)



b) Concentration Profile for Gypsum Wallboard, VHP Exposure at 125-150 ppm (31May05 run)

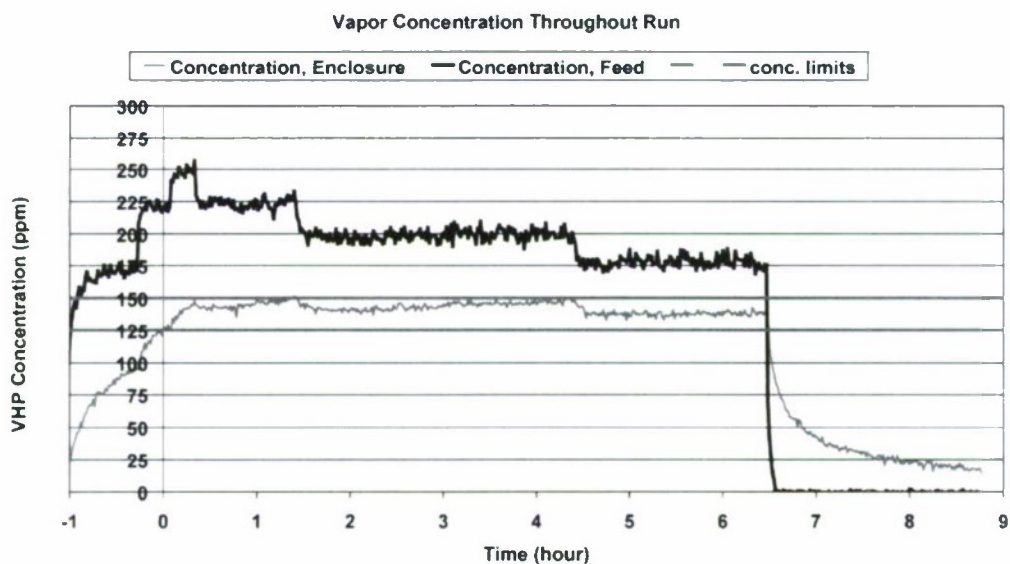
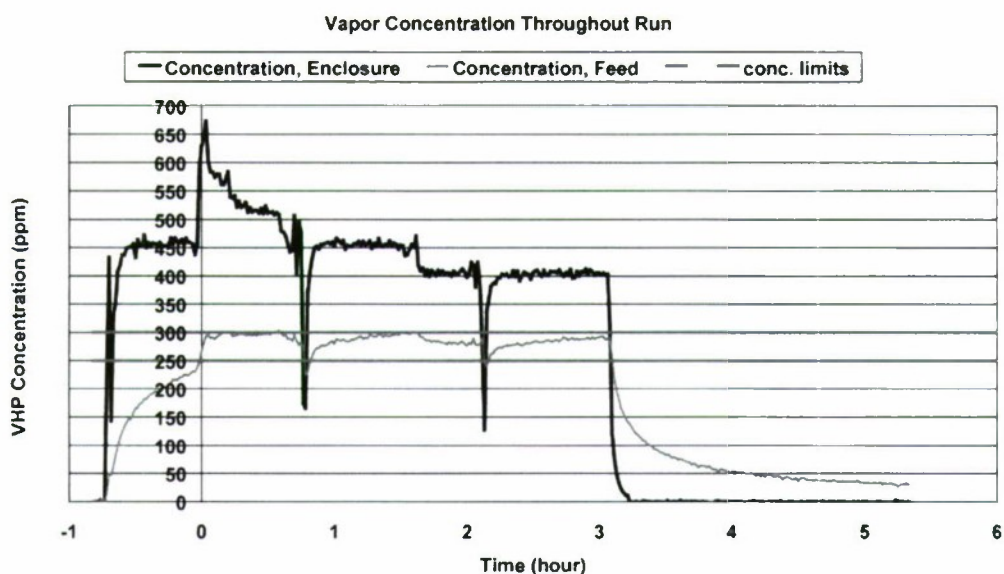


Figure 18. Representative concentration profile results for gypsum wallboard.

a) Concentration Profile for Acoustical Ceiling Tile, VHP Exposure at 250-300 ppm (16Mar05 run)



b) Concentration Profile for Acoustical Ceiling Tile, VHP Exposure at 125-150 ppm (1Jun05 run)

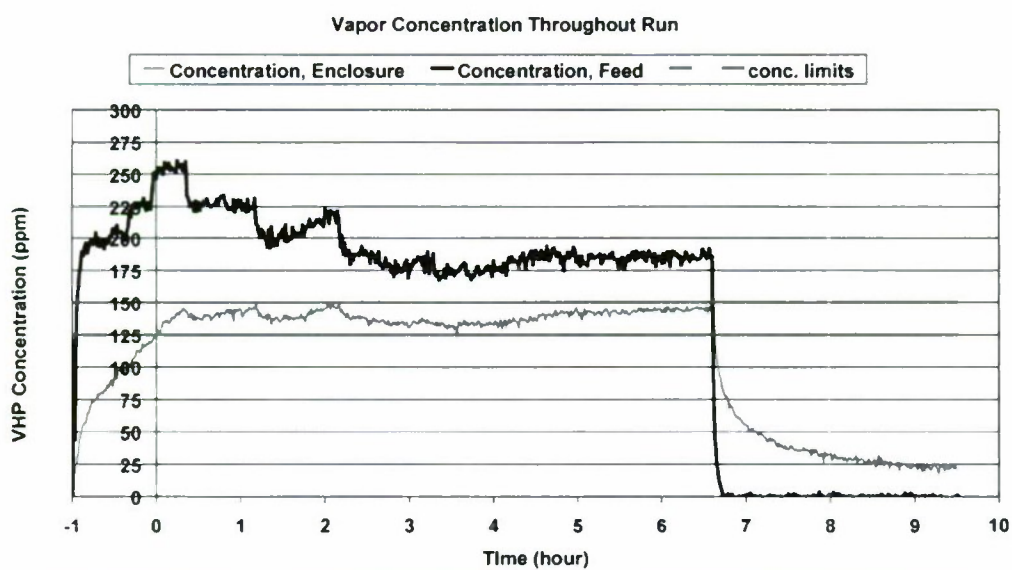
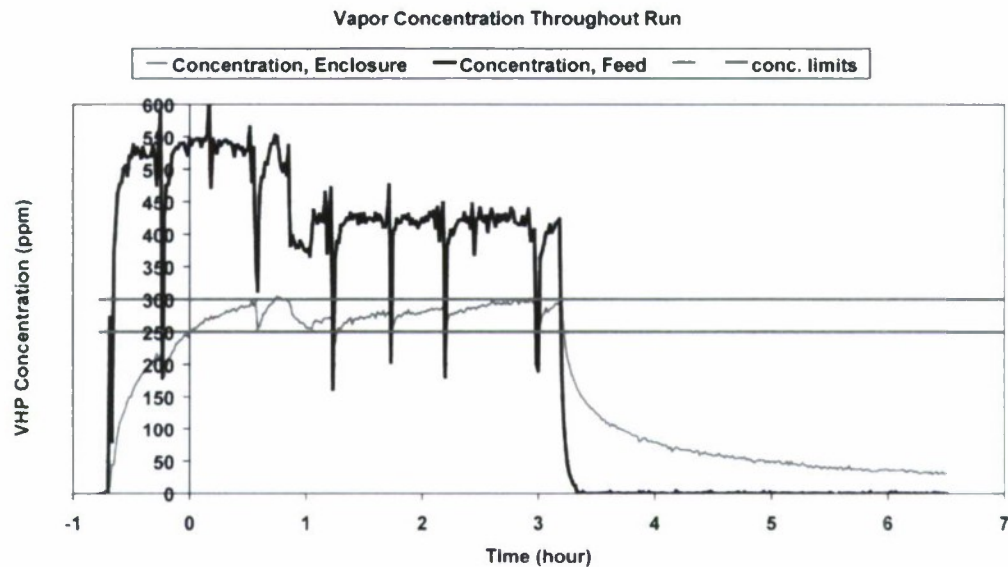


Figure 19. Representative concentration profile results for acoustical ceiling tile.

a) Concentration Profile for Wood, VHP Exposure at 250-300 ppm (26Apr05 run)



b) Concentration Profile for Wood, VHP Exposure at 125-150 ppm (10May05 run)

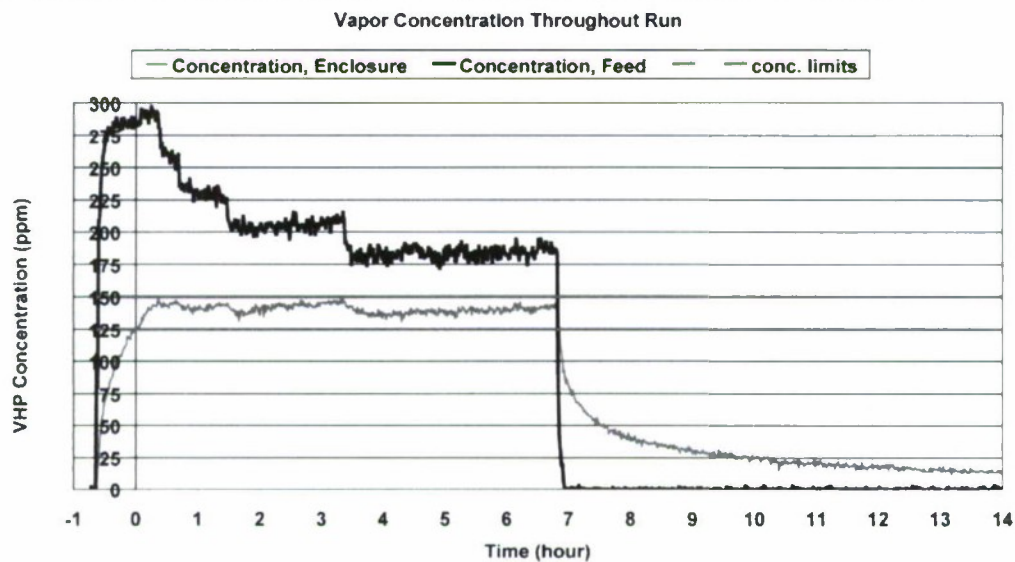
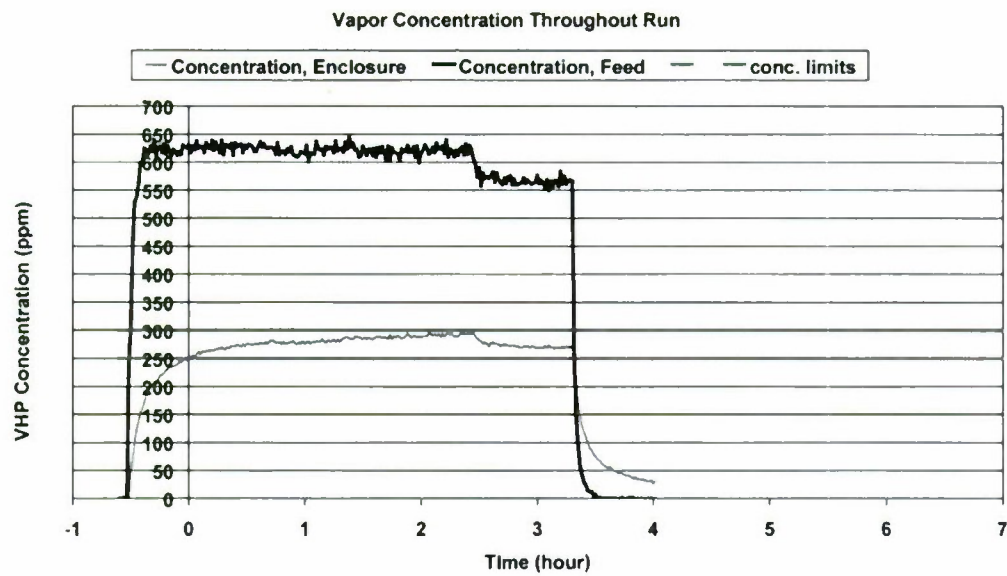


Figure 20. Representative concentration profile results for wood.

a) Concentration Profile for Concrete Cinder Block, VHP Exposure at 250-300 ppm (4May05 run)



b) Concentration Profile for of Concrete Cinder Block, VHP Exposure at 125-150 ppm (2Jun05 run)

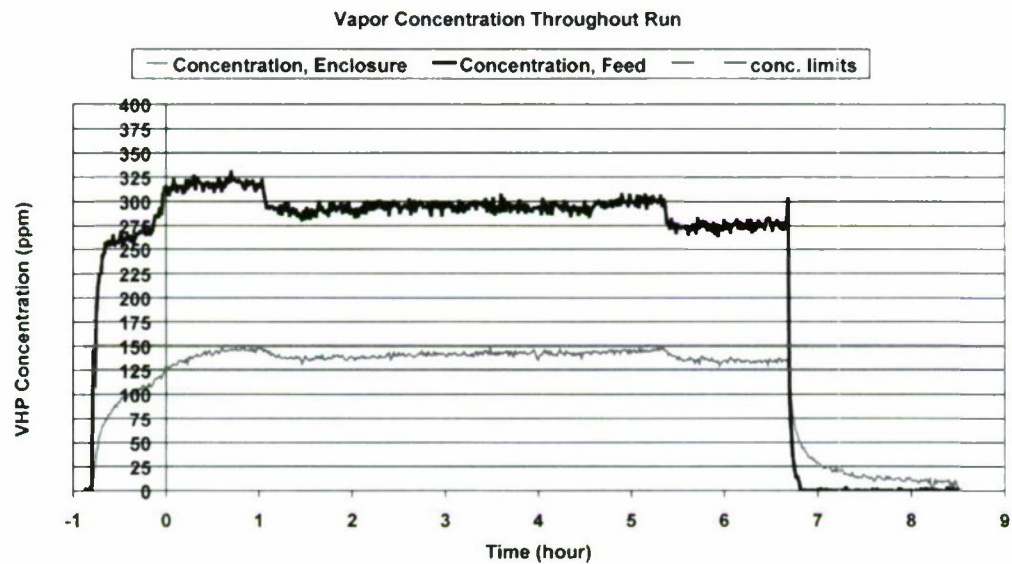


Figure 21. Representative concentration profile results for concrete cinder block.

6.2

Discussion

Carpet and the two painted materials (steel and wallboard) had the lowest VHP material demand. The sorptive building materials, such as ceiling tile, wood, and concrete cinder block had greater VHP material demand. Of the materials studied, concrete cinder block showed the greatest material demand.

Using the baseline as the reference point, some of the materials adsorbed VHP and others decomposed VHP. The aeration time for the 250 ppmv VHP run is shown in Figure 22a. Figure 22b shows a close-up view of the aeration cycle at the 30 ppmv end of run. Bars highlight the aeration span covered by the three replicate runs for each material and the empty chamber.

The concrete cinder block required the greatest increase in generator output to maintain the target concentration within the enclosure, hence the highest material demand. The concrete cinder block also had the shortest aeration time, indicating that the majority of the excess VHP introduced into the chamber was decomposed by the concrete cinder block surface.

The two cellulose-based materials, wood and acoustical ceiling tile, required an increase in the generator output to maintain the target concentration within the enclosure. The wood and acoustical ceiling tile tests also had the longest aeration time, indicating that these materials adsorbed VHP during the decontamination phase and off-gassed VHP during the aeration phase.

The wallboard test results had a similar material demand value and shorter aeration time, compared with ceiling tile and wood. Based on this comparison, the VHP was most likely adsorbed and decomposed by the painted wallboard surface.

The carpet test results indicated a low material demand value and an aeration time similar to the baseline study. Based on this comparison, the VHP was not adversely affected by the carpet.

The steel test results indicated a low material demand value compared to wallboard, ceiling tile, wood, and concrete cinder block. The steel samples also had a short aeration time compared with the baseline tests. The steel may be adsorbing or decomposing the VHP.

Based on these results, building materials may impact the ability to maintain the target concentration by adsorption and/or decomposition of the VHP. In addition, some materials may continue to offgas VHP after decontamination is completed, resulting in longer cycle times.

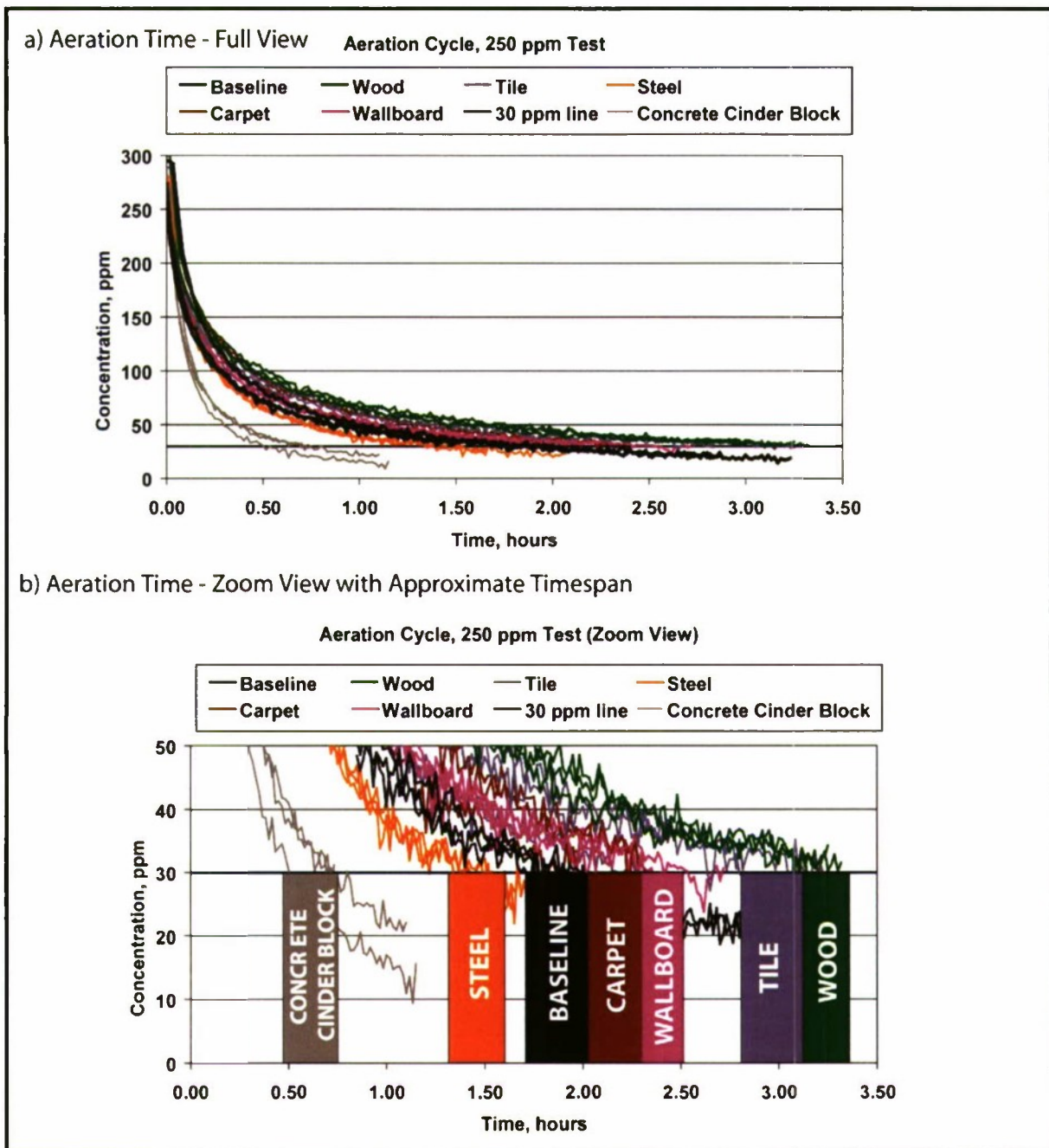


Figure 22. Aeration time for building materials exposed to 250-ppmv VHP.

6.3 Consequences for Building Decontamination

This study provides information that may be used as guidance for the VHP concentration and aeration time requirements when decontaminating an interior space containing the building materials studied. Based on these results, building materials may impact the ability to maintain the target concentration by adsorption and/or decomposition of the VHP. In addition, some materials may continue to offgas VHP after decontamination is completed, resulting in longer cycle times.

The material demand values reported can be used to estimate the total hydrogen peroxide capacity required to maintain the desired peroxide concentration. For example, consider two 900 m² (~10,000 ft²) spaces—one a warehouse type environment and the other an office space. This hypothetical warehouse will have a concrete floor, cinderblock walls, and a steel roof. The office building will have a carpeted floor, painted wallboard walls, and a dropped ceiling. In both buildings, the walls are 3 m from the floor to the ceiling/roof, and the building is 30 m on each side. The buildings will be fumigated at a concentration of 300 ppmv of hydrogen peroxide for 3.33 h, resulting in a CT of 1000 ppmv-h.

Table 6 and Table 7 give the estimated hydrogen peroxide production capacity by multiplying the surface area of each material present in the building by the flux, J, from Table 5. The calculated requirement represents the excess capacity the hydrogen peroxide generator must supply, in addition to that needed to maintain a concentration of 300 ppmv, or 0.42 g/m³. To illustrate how this information could be used in practice, consider the STERIS VHP® 100M, which in an open loop configuration has maximum peroxide and airflow rates of 504 g/h (24 g/min of 35% H₂O₂) and 75 m³/h, respectively, for a feed concentration of 6.7 g/m³. Of that feed concentration, 6.3 g/m³ would be available to overcome the material demand in the contaminated space. Dividing the material demand by this excess generation capacity results in the air-exchange rate required to maintain the air concentration of peroxide and overcome the material demand.

Table 6. Material demand of warehouse surfaces.

Surface	Surface area (m ²)	J (g/hr/m ²)	HP required (g/hr)	Air exchange (m ³ /hr)
Concrete floor	929	3.61	3354	532
Cinderblock walls	360	3.61	1300	206
Steel ceiling	929	0.455	423	67
Total			5076	805

Table 7. Material demand of office surfaces.

Surface	Surface area (m ²)	J (g/hr/m ²)	HP required (g/hr)	Air Exchange (m ³ /hr)
Carpet	929	0.338	314	50
Painted Wallboard	360	1.403	505	80
Ceiling tile	929	1.650	1532	243
Total			2351	373

From Table 6 and Table 7, it is clear that the materials within a particular building can have a significant impact on the generation capacity required. The concrete/cinderblock building requires about twice as much vapor generation capacity as the office space, mainly due to the high material demand of the concrete. As a result, the warehouse would require, at a minimum, eleven STERIS VHP® 100M units, while the office building would require at least five. These requirements could be lowered if the air circulation, temperature, and relative humidity within the buildings were such that the hydrogen peroxide vapor generators could be configured to output at even higher concentrations.

7. QUALITY ASSURANCE FINDINGS

Four technical systems audits were completed. Overall there were no follow-up corrective actions from testing. During the first audit, two on-the-spot corrections were made. The first correction was during the chamber shutdown procedure. The operator skipped a step in the procedure. The auditor caught the mistake and the operator went back and completed the shutdown. The second mistake was the misnumbering of one coupon. The operator identified the error, measured the coupon again, and matched it to the correct CoC card. The sample was renumbered. The corrections during the first audit were not unexpected because the procedures and equipment were new, and the audit was done during the first test.

8. CHALLENGES AND LESSONS LEARNED

8.1 VHP Relative Humidity Sensors

The Vaisala HUMICAP humidity sensor (model HMT333) reading was slightly high in the presence of VHP at approximately 300 ppmv. But when the sensor was removed from air containing VHP, the sensor responded normally with no change in response. The relative response of the sensor to VHP and humidified air was checked by separately injecting distilled water or 35% hydrogen peroxide at identical rates into the VHP generator and observing the resultant %RH in the chamber after equilibration. The injection rate was similar to that used during most of the exposure runs. When distilled water or 35% hydrogen peroxide was injected, the sensor read 32.2 and 34.3%RH, respectively, indicating that the sensor read the %RH high by approximately 2%RH units in the presence of VHP. Even though the VHP effect on %RH was small, it did not have any impact on the maximum 30%RH SOR requirement because the sensor was not exposed to VHP at the SOR. The %RH probe response was verified, after completing approximately 3/4 of the runs, by exposure to humidified air above saturated salt slurries. Slurries of potassium acetate, potassium carbonate, and sodium chloride were used to yield standard humidities of 23, 44, and 76%RH respectively. The variations between the standard and sensor %RH readings were less than 3.5% (23.4, 42.6, and 77.9%RH, respectively). During test runs, the %RH mimicked the VHP concentration in the enclosure, showing that the change in %RH was generally related to the changes in VHP concentration (Figure 23).

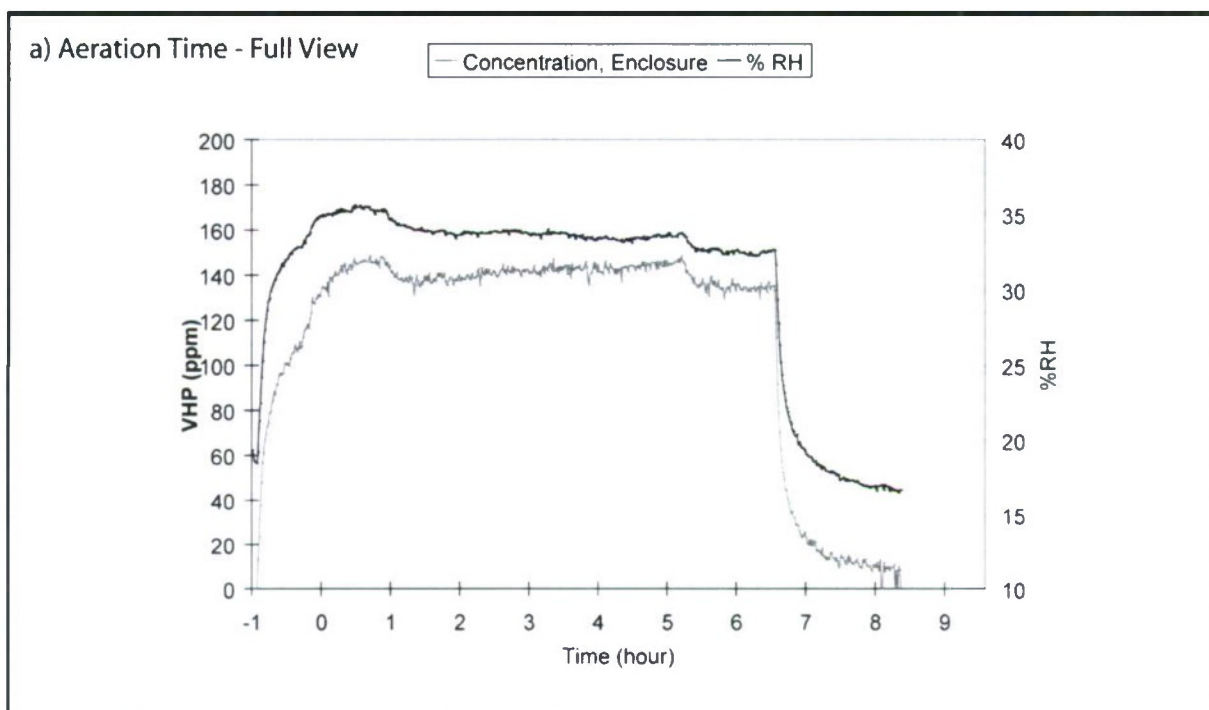


Figure 23. Comparison of VHP concentration and %RH.

8.2 Calibration of Dräger Hydrogen Peroxide Electrochemical Sensors

Initial attempts to calibrate the hydrogen peroxide sensors using sulfur dioxide in nitrogen gas revealed that the sensors were highly sensitive to changes in pressure. The calibration procedure with sulfur dioxide required the removal of the sensors from their test enclosures. The sensors were placed in a low volume calibration adaptor to allow the passage of the calibration gas. This conserved the calibration gas and enabled quick equilibration of the calibration gas concentration. The calibration procedure was quick and straightforward. But when the sensors were placed in their test enclosures, the inlet sensor reading was higher than the enclosure sensor, due to slightly more pressure in the inlet enclosure than the exit enclosure. Switching the sensors revealed that the variation was due to their placement, and not due to the specific sensor. The sensors could be calibrated with the sulfur dioxide gas procedure, but the verification agreement with the peroxide concentration from the iodometric titration was typically in error by approximately 15% due to the slight variation in enclosure pressures. Attempts to refine the sulfur dioxide calibration procedure and successfully validate were exhausted.

The hydrogen peroxide sensors were recalibrated in place using VHP concentration values, determined by chemical titration of VHP captured in bubbler solutions. The inlet hydrogen peroxide detector was calibrated to measure from 0 to 800 ppmv H_2O_2 , and the outlet hydrogen peroxide detector was calibrated to measure from 0 to 340 ppmv H_2O_2 IAW IOP DS04001. The outlet sensor was expected to detect concentrations no greater than 300 ppmv, and was, therefore, calibrated slightly at 340 ppmv. The inlet sensor was calibrated from 0 to 800 ppmv H_2O_2 to accommodate anticipated higher VHP concentrations in the feed air. Verification of both sensors was conducted during each run, using the average value from three iodometric titrations on the VHP stream entering and exiting the glove box (IOP DS04019). This calibration and verification procedure was similar to that used for the chlorine dioxide system.⁷

Blank

LITERATURE CITED

- (1) Jahnke, M.; Lauth, G., Biodecontamination of a Large Volume Filling Room with Hydrogen Peroxide, *Pharm. Eng.* **1997**, 2-12.
- (2) McDonnell, G. G.; Gringol, G.; Antloga, K. Vapour-Phase Hydrogen Peroxide Decontamination of Food Contact Surfaces, *Dairy Food Environ. Sanit.* **2002**, 868-873.
- (3) Brickhouse, M. D.; Turetsky, A.; MeVey, I. *Decontamination of CBW Agents by mVHP: Demonstration of the CBW Decontamination of a Building using mVHP*; ECBC-TR-740; Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2005; UNCLASSIFIED Report (AD-B333 526).
- (4) Brickhouse, M. D.; Turetsky, A.; Maelver, B.; Pfarr, J.; Dutt, D.; MeVey, I.; Alter, W.; Lloyd, J. P.; Mark A. Fonti, J. *Vaporous Hydrogen Peroxide Decontamination of a C-141B Starlifter Aircraft: Validation of VHP and mVHP Fumigation Decontamination Process Via VHP-Sensor, Biological Indicator, and HD Simulant in a Large-Scale Environment*; ECBC-TR-510; Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2005; UNCLASSIFIED Report (AD-A468 145).
- (5) *EPA Guidance on Environmental Data Verification and Data Validation*, EPA QA/G-8, U.S. Environmental Protection Agency, Washington, DC, 2002.
- (6) Wagner, G. W.; Sorrick, D. C.; Procell, L. R.; Hess, Z. A.; Brickhouse, M. D.; MeVey, I. F.; Schwartz, L. I. In *CB Defense*: Timonium, MD 2003.
- (7) Brickhouse, M. D. *Quality Assurance Project Plan and Work Plan for Effects of Vaporized Decontamination Systems on Selected Building Interior Materials*; Doc. No. DSQAPP2004MC, Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2004.
- (8) *Quality Management Plan (QMP) for the National Homeland Security Research Center (NHSRC) Office of Research and Development (ORD)*, U.S. Environmental Protection Agency, Washington, DC, 2003.
- (9) *Quality Management Plan for Environmental Programs*, Edgewood Chemical Biological Center, Research, Development, and Engineering Command: Aberdeen Proving Ground, MD, 2003.
- (10) *EPA Guidance for Data Quality Assessment. Practical Methods for Data Analysis*, EPA QA/G-9, U.S. Environmental Protection Agency, Washington, DC, 2000.
- (11) *EPA Requirements for Quality Assurance Project Plans*, EPA QA/R-5, U.S. Environmental Protection Agency, Washington, DC, 2001.
- (12) *EPA Guidance for Quality Assurance Project Plans*. EPA QA/G-5, U.S. Environmental Protection Agency, Washington, DC, 2002.
- (13) Lalain, T. *Effects of Vaporized Hydrogen Peroxide on Selected Building Interior Materials*; ECBC-TR-661; Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2009; UNCLASSIFIED Report (AD-A496 258).

(14) Procell, L. *Material Exposure to Vaporized Hydrogen Peroxide*; RNG-107, Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2005.

(15) Procell, L. *Procedure for the Operation of the Hydrogen Peroxide Glove Box and Exposure of Materials*; DS04015, Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2005.

(16) Lalain, T. *Placement of Coupons in Chambers for Deposition Velocity / Material Compatibility Testing for the EPA Program*; DS04016, Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2005.

(17) Brickhouse, M. D. *Quality Assurance Project Plan and Work Plan for Deposition Velocity Studies: Materials Sorption of Vaporized Hydrogen Peroxide or Chlorine Dioxide*; Doc. No. DSQAPP2004DV, Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2004.

ACRONYMS

A	Surface area
APG	Aberdeen Proving Ground
ASTM	American Society for Testing and Materials
Avc	Average
CB	chemical and biological
CoC	chain-of-custody
CFM	Cubic Feet Minute
CT	concentration multiplied by time
CT _{inlet}	CT of the affluent
CT _{diff,b}	Difference in CT _{inlet} and CT _{outlet} for the baseline tests
CT _{diff,k}	Difference in CT _{inlet} and CT _{outlet} for a specific material after baseline correction
CT _{outlet}	CT of the effluent
d _{H₂O₂}	density (g/L) of H ₂ O ₂ at 30 °C using PV = nRT
DS	Decontamination Sciences
ECBC	Edgewood Chemical and Biological Center
EOR	end of run
EPA	U.S. Environmental Protection Agency
F _{total}	Total Volume defined by test limits (0 – 1000 ppmv-h)
H ₂ O ₂	hydrogen peroxide
IAW	in accordance with
IOP	Internal Operating Procedure
IS	internal standard
ISO	International Standardization Organization
J	mass flux
mb	Specific Material
MD	Material Depend
M _{H₂O₂}	Mass of hydrogen peroxide
NHSRC	National Homeland Security Research Center
P _{sys}	chamber pressure
PPB	part-per-billion
PPMV	part-per-million by volume
QA	quality assurance
QAPP	Quality Assurance Project Plan (QAPP)
QMP	Quality Management Plan
RH	relative humidity
SOPs	standing operating procedures (standard may also be used in place of standing with the same meaning)
Std dev	Standard deviation
SOR	start of run
TICs	toxic industrial chemicals
TIMs	toxic industrial materials
TR	technical report
TSA	technical systems audit
Ver	version
VHP [®] , VHP	Steris' registered "vaporized hydrogen peroxide" procedure

COUPON SPECIFIC CODING

"W"	bare wood
"R"	carpet
"T"	ceiling suspension tile
"G"	latex-painted gypsum wallboard
"S"	painted structural A572 steel
"C"	unpainted concrete cinder block

APPENDIX A

DETAILED COUPON PREPARATION AND INSPECTION PROCEDURES

COUPON PREPARATION PROCEDURE

The coupon preparation, unless otherwise noted, will be conducted at the Edgewood Chemical Biological Center Experimental Fabrication Shop.

Mechanically Graded Lumber (Bare Wood)

- Stock Item Description: 2 x 4 x 8 KD WW/SPF Stud
- Supplier/Source: Home Depot, Edgewood Maryland
- Coupon Dimensions: 10 x 1½ x ½ in.
- Preparation of Coupon:
 - The machined ends of the stock will be discarded by removing >¼ in. of the machined end. Coupons will be cut from stock using a table saw equipped with an 80 tooth crosscut blade.

Latex-Painted Gypsum Wallboard

- Stock Item Description: ½ in. x 4 ft. x 8 ft. Drywall
- Supplier/Source: Home Depot, Edgewood Maryland
- Coupon Dimensions: 6 x 6 x ½ in.
- Preparation of Coupon:
 - The ASTM method requires that the samples be taken from the interior of material rather than from the edge (machined edge). The machined ends of the stock will be discarded by cutting away >4 in. from each side.
 - Coupons will be cut from stock using a table saw equipped with an 80 tooth crosscut blade.
 - The 6 x 6 in. coupons will be painted with 1-mil of Glidden PVA Primer and followed by 1 to 2-mils of Glidden latex topcoat. The primed coupons will be allowed to stand for >24 h prior to the application of the topcoat.
 - All six sides of the 6 x 6 in. coupon will be painted.

Concrete Cinder Block

- Stock Item Description: 8 x 16 x 1.5 in. concrete block cap
- Supplier/Source: York Supply, Aberdeen Maryland
- Original Coupon Dimensions: 4 x 8 x 1.5 in.
- Modified Coupon Dimensions: 4 x 8 x 0.5 in.
- Preparation of Coupon:
 - Coupons will be cut from stock using a water-jet.
 - Four coupons will be cut from each stock piece.
 - Original dimensions were too large for material testing:
 - Each coupon was cut into three sections.
 - Two sections were measured at modified coupon dimensions.
 - Third section was discarded.

Carpet

- Stock Item Description: 12 ft Powerhouse 20 Tradewind
- Supplier/Source: Home Depot, Edgewood, Maryland
- Coupon Dimensions: 6 x 8 in.
- Preparation of Coupon:
 - Coupons will be cut from the stock using a utility knife.
 - The longer direction (8 in.) will be cut parallel to the machine edge.
 - The machined edge will be discarded by removing $> \frac{1}{2}$ in.

Painted Structural Steel

- Stock Item Description: A572 Grade 50, 4 ft x 8 ft. x $\frac{1}{4}$ in.
- Supplier/Source: Specialized Metals
- Coupon Dimensions: $\frac{1}{4}$ x 12 in. total, dog bone shaped with 2 in. wide at ends, $\frac{3}{4}$ in. wide center
- Preparation of Coupon:
 - Coupons will be cut from stock using a water jet.
 - A visual observation will be conducted on each coupon to determine if size and shape have deviated from the required dimension, and discarded if rejected.
 - Coupons will be cleaned and degreased following procedures outlined in TTC-490.
 - Coupons prepared for painting per TT-P-645 with red oxide primer.

The ECBC Experimental Fabrication Shop prepared the materials IAW the standards used for the preparation and painting of steel. TTC-490 is a Federal Standard providing cleaning methods and pretreatment of iron surfaces for the application of organic coatings. The pretreatment is the application of a zinc phosphate corrosion inhibitor. TT-P-645 is a Federal Standard for the application of alkyd paint. These standards were not obtained through this program, but were purchased by the Shop for their work.

Ceiling Suspension Tile

- Stock Item Description: Armstrong 954, Classic Fine Textured, 24 x 24 x $\frac{9}{16}$ in.
- Supplier/Source: Home Depot, Edgewood, Maryland
- Coupon Dimensions: 12 x 3 x $\frac{9}{16}$ in.
- Preparation of Coupon:
 - Coupons will be cut from stock using a table saw equipped with an 80-tooth crosscut blade.
 - Sixteen samples will be removed from each stock item.

COUPON INSPECTION PROCEDURE

All coupons will be inspected prior to testing to ensure that the material being used is in suitable condition. Coupons will be rejected if there are cracks, breaks, dents, or defects beyond what are typical for the type of material. In addition, coupons will be measured to verify the dimensions. Coupons deviating from the dimension ranges listed as follows will be discarded:

Mechanically Graded Lumber (Bare Wood)	$10 \pm 1/16 \times 1.5 \pm 1/16 \times 0.5 \pm 1/32$ in.
Latex-Painted Gypsum Wallboard	$6 \pm 1/16 \times 6 \pm 1/16 \times 0.5 \pm 1/16$ in.
Concrete Cinder Block	$4 \pm 1/2 \times 8 \pm 1/2 \times 0.5 \pm 1/8$ in.
Carpet	$6 \pm 1/8 \times 8 \pm 1/8$ in.
Painted Structural Steel	$1/4 \pm 1/128 \times 12 \pm 1/16$ in., with $2 \pm 1/16$ in. wide at ends, $3/4 \pm 1/16$ in. wide in the center Lot SS: same but with 0.27 ± 0.02 in. thickness
Ceiling Suspension Tile	$12 \pm 1/8 \times 3 \pm 1/16 \times 9/16 \pm 1/16$ in.

Blank

APPENDIX B

COUPON IDENTIFIER CODE

All coupons will be marked with an ID number that will consist of a nine-character alphanumeric code. A description of the identifier pattern and an example code are shown below. Figure 24 shows the coupon placement in the chambers.

Code Pattern

<u>Character</u>	<u>Explanation</u>
1	Material W = wood G = gypsum S = A572 steel T = acoustic ceiling tile C = concrete cinder block R = carpet B = circuit breakers
2	Fumigant: V = VHP N = no fumigant
3	Test start date
4, 5	year for example: 4 = 2004
6, 7	month for example: 06 = June
	day for example: 10 = the 10 th of a month
8, 9	Chamber position (see IOP DS04016 figure 1)

Example

GV4101104

Gypsum Wallboard, with test start date of October 11th, 2004, is sample number 4.

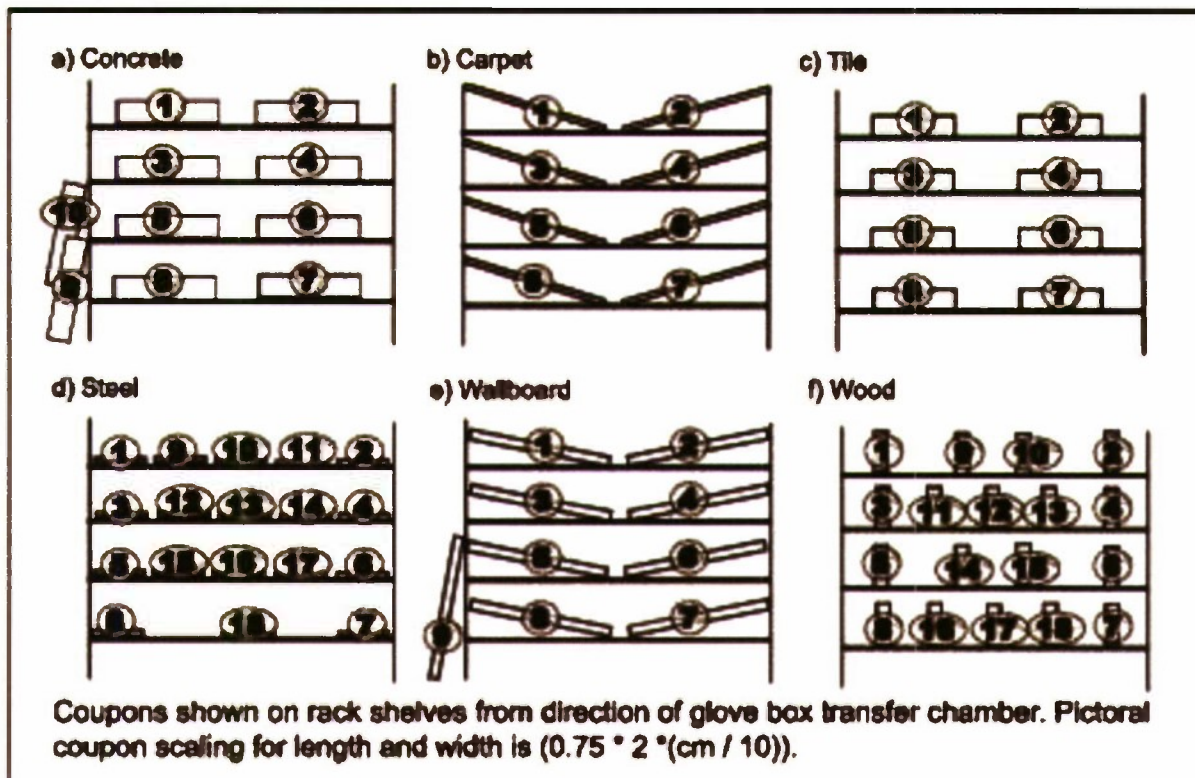


Figure 24. IOP DS04016 Coupon placement in chambers.